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SOFT WINTER WHEAT STUDIES. IV. SOME FACTORS PRODUCING VARIATIONS IN WHOLEMEAL "TIME" DATA

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During the past three or four years the evaluation of soft winter wheats by short methods has received much additional attention by soft wheat mill chemists. This increased interest has been due in part to the published fermentation method of Saunders and Humphries (1928), the *Schrotgärmethode* of Pelshenke (1930), and the wheat-meal fermentation time test of Cutler and Worzella (1931). The latter two tests employ whole-wheat meal whereas the first mentioned is a flour test. All three are similar in that they are fermentation tests made upon doughs employing only a leavening agent (yeast) and water. No sugar, salt, or other materials are added as in the case of the baking test.

To plant breeders engaged in evaluating new wheats, the tests using wheat meal have proven of great interest as these tests require but small amounts of grain per determination. Winter and Gustafson (1934), Wilson and Markley (1934), and Worzella (1934) have employed the Cutler-Worzella method in their testing of variety and breeding samples in this country. Cutler and Worzella (1933) have proposed a classification of world wheats using " time " as an index. They suggest their test may prove useful in making blends in the mill.

The Pelshenke procedure, in addition to being extensively used in Germany by its originator, has been favorably commented upon by Edel (1934) working largely with European wheats, while Griffiths and Cayzer (1934), Breakwell (1934), and Hickinbotham (1934, 1934a) find the test reasonably reliable with Australian wheats.

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[2] Presented Feb. 16, 1935 before the Cincinnati Division of the Central States Section as a contribution to a discussion on the whole meal fermentation time test.

The author (Bayfield, 1934) working with Ohio wheats has recently shown that the 10-g. doughball of Cutler and Worzella gave definitely different results from those obtained when using the 5-g. doughball specified by Pelshenke. The two methods ranked samples in different orders. Relative strength ratings of different varieties were changed depending upon the method used. Size of doughball *relative* to the diameter of the vessel used for the test appears to be the major difference between the two methods. With the former method *strong* wheat samples had their times prolonged more or less due to the walls of the vessel supporting the fermenting dough. Both Pelshenke (1930) and Hickinbotham (1934) have observed that doughballs thus supported give longer times. The method of Pelshenke (1930) avoids this difficulty by using a doughball much smaller *relative* to the diameter of the beaker (5-g. meal per ball, 6 cm. diameter of vessel) than is specified in the Cutler-Worzella method (10-g. meal per ball, diameter of beaker approximately 5.4 cm., *i.e.*, low form 150 cc. size).

The author obtained very variable results in using the 10-g. doughballs when samples possessed strengths of such a nature that some of the samples were supported more or less by the beaker but not in all replicates. The author believes that the " time " variations in variety No. 912203 obtained by Winter and Gustafson (1934, page 51) may be explained on this basis.

Method and Material

For complete details of grinding samples and performing the test the reader is referred to the earlier paper by the author (Bayfield, 1934). Briefly, the samples were gound on a Wiley mill and, unless otherwise specified, all tests were carried out at 30 $\pm$ 0.2° C. using 150 cc. low form beakers with 80 cc. distilled water. Fresh 10% (10 g. Fleischmann yeast in 100 cc. water) yeast suspension was made up daily and used after standing at 30° C. for at least 30 minutes. Each 10 g. of meal received 5.5 cc. of this suspension. Smaller doughs received proportionately less.

The method of grinding employed by the author probably produces a meal differing materially from that used by Pelshenke who used a grinder of European manufacture. Pelshenke (1933) points out that disk mills are unsatisfactory for the grinding of the meal on account of the heat generated in grinding and also because of the danger of dead grinding. C. O. Swanson (personal communication, 1934) states that he finds the Wiley mill preferable to other mills studied. Wilson and Markley (1934) state that the meals used by Pelshenke were ground so that the endosperm was reduced to essentially the fineness of flour. Chapman and Farquhar (1934) compared meals ground on a

trituration mill with meals ground in a coffee mill. The former type of grinder gave higher time figures. Apparently the coffee mill is used extensively for these tests in Australia.

As several workers have already observed, *scrupulous* care to small details is necessary in the performance of the test if it is expected to duplicate results satisfactorily. It may therefore be anticipated that it will be difficult for different laboratories to check each other's results. As a result of various inconsistencies in data it was decided to investigate a few of the factors which seemed to be influencing these results.

Experimental

Vessel size and volume of water. The Cutler-Worzella procedure (1933) stipulates a proportionate reduction in amount of yeast, size of beaker, and volume of water used in beaker, when the doughball is reduced from the recommended 10-g. size. Table I indicates that

TABLE I

EFFECT OF VESSEL DIAMETER AND VOLUME OF WATER UPON TIME
(13.8% protein Red Rock wheat, averages of 10 tests)

Amount water used	Vessel diameter	Time 10-g. meal	Time 5-g. meal
Cc.	*Cm.*	*Min.*	*Min.*
	Varying volume of water		
80	5.3	205[a]	83
120	5.3	177[a]	81
200	5.3	173[a]	76
	Varying diameter of vessel		
80	5.4	202[a]	85
120	6.6	85[b]	87
200	7.4	76	84

[a] Time prolonged by vessel walls supporting dough mass.
[b] One replicate partially supported by vessel walls.

increasing the diameter of the beaker causes large changes in time with this size of doughball. Varying the amount of water alters the time but not as extensively as when the size of vessel is changed.

Table I presents a possible explanation for the wide difference in range of time for world wheats obtained by the two methods. Thus Pelshenke (1930) reports his strongest sample (Northern Duluth) at 282 minutes whereas Cutler and Worzella (1931) found North Dakota Marquis wheat had a time of 391 minutes. With a Canadian hard red spring wheat (15.8% protein) the author obtained a time of 387 minutes with the 10-g. doughball while the same sample of meal when tested so that no support was given by the vessel walls gave a time of 221 minutes.

Varying size of doughball. It should be pointed out that throughout this paper the weight, *e.g.*, 10- or 5-g. doughballs, refers to the amount of *meal* used in a dough. The weight of the mixed dough naturally would be heavier on account of added yeast and water.

To obtain further information regarding size of doughball a number of tests were run. A series of wheats consisting of 3 varieties each grown at the same 20 locations in 1933 were time tested in quadruplicate by both the 10- and 5-g. doughballs. The varieties were Red Rock (a " strong " soft red winter), Trumbull (medium strength soft red winter), and American Banner (weak white winter). During these tests observations were made to note samples receiving support from the vessel walls. Table II gives the resulting data.

TABLE II

A COMPARISON OF 10- AND 5-G. DOUGHBALLS

(Average results from 20 samples of each variety)

	Variety			Average 60 samples
	Red Rock	Trum-bull	American Banner	
Wheat protein—%	11.2	11.0	10.4	10.9
Loaf volume (regular)—*Cc.*	623	607	563	597
Time—10-g.—*Min.*	133	57	33	74
5-g.—*Min.*	65	51	34	50
Number of samples supported by vessel				
10-g.	15	2	0	
5-g.	0	0	0	

Table II shows the large influence which the vessel wall exerts upon time with stronger samples. Examination of the data for the individual samples also showed that where the vessel was not interfering with the time the smaller sized doughballs gave the longer times. The 18 Trumbull samples which did not receive support from the vessel averaged 46 minutes for the 10-g. tests and 50 minutes in 5-g. tests. Of the 2 Trumbull samples which were influenced by the vessel walls when tested by the 10-g. doughballs, one equaled 153 and the other 168 minutes. The same two samples according to the 5-g. doughs gave times of 55 and 61 minutes, respectively.

Table III presents data which further indicate that the smaller sized doughballs tend to give the longer times when the vessel size is not a disturbing factor. The data also show that individually mixed doughballs give times which are different from " twin " doughballs formed by halving a single larger dough. It will be observed that the 2.5-g. twin doughballs give longer times than 5-g. twins.

TABLE III

COMPARISON OF TWO METHODS OF MAKING DOUGHBALLS

Sample No.	Variety	Wheat protein	Loaf volume (bromate)	Time in minutes		
				Undivided 5-g. doughs	10-g. doughs divided in half	5-g. doughs divided in half
		%	*Cc.*			
1571	Trumbull	12.2	590	39	39	43
1572	Nabob	12.1	552	48	42	45
1573	Fulhio	12.9	575	32	29	31
1574	Red Rock	12.8	677	62	59	69
1575	American Banner	11.4	562	30	24	24
1576	Bald Rock	12.9	612	57	47	54
1577	Michigan Amber	13.7	630	53	51	62
1578	Kharkov	12.8	580	36	33	39
1579	Fultz	12.3	530	33	27	35
1580	Gladden	12.7	620	35	31	42
	Average	12.58	592.8	42.5	38.2	44.4

Effect of varying granulation. An extensive series of grindings were made in preliminary experiments on wheats ranging in strength from Michikof to American Banner before deciding upon the grinding procedure finally adopted. The Wiley mill at the start of these experiments was new and the distances separating the various grinding knives were quite variable. A series of 10 different varieties ground on the mill thus set averaged 49.8 minutes (5 g. meal). The same series when ground with the knives set with a clearance of 0.009–0.010 inches (standard setting adopted) averaged 53.0 minutes. The standard ground meal was by appearance slightly finer.

To test further the effect of granulation, a 13.8% protein Red Rock sample was ground using the three screens provided with the mill. The largest size sieve gave a very coarse meal and the finest screen a meal in appearance like a very granular low grade flour. In forming the doughballs the medium ground meals handled much more satisfactorily than either the finer or coarser material. Table IV presents the resulting data.

TABLE IV

EFFECT OF VARIOUS WILEY SIEVES UPON TIME

Sieve used	0.5 mm.	1.0 mm.	2.0 mm.
Time in minutes			
10-g. doughballs	over 300 [1]	202	164
5-g. doughballs	243	101	111

[1] Dough not entirely down after 18 hours.

This test showed that the coarsest meal with the 10-g. doughball received support from the beaker wall, thus prolonging the time. The very coarse meal 5-g. doughs seemed to receive some support as well, although this did not occur with the other 5-g. doughballs. These coarse meal doughs spread over the surface of the water in a surprising manner. The results confirmed the conclusion of Cutler and Worzella (1931) that a medium fine meal is best for the test. Chapman and Farquhar (1934) found the meals from a coffee mill preferable to the much finer meals obtained by grinding on a trituration mill.

Effect of heat treating grain. Kent-Jones (1927) has shown that heating wheat strongly increases its strength when this heating is carried out under certain specified conditions. A number of residues of samples which had been held at 140° F. for several days in an insect eradication campaign were available for testing to see if this treatment affected time. A high (average 12.1%) and a low (average 9.6%) protein series were run, each series consisting of the same 10 varieties. Table V gives the results from these 5-g. doughball tests.

TABLE V

EFFECT UPON TIME OF HEAT-TREATED GRAIN

Variety	Low protein series			High protein series		
	Not heated	Heated	Increase	Not heated	Heated	Increase
	Min.	*Min.*	*Min.*	*Min.*	*Min.*	*Min.*
Trumbull	47	60	13	81	98	17
Nabob	44	55	11	91	162	71
Fulhio	39	50	11	74	145	71
Red Rock	50	64	14	110	109	−1
American Banner	27	27	0	51	69	18
Bald Rock	55	63	8	86	82	−4
Michigan Amber	45	70	25	96	113	17
Kharkov	39	45	6	67	106	39
Fultz	44	61	17	83	112	29
Gladden	34	41	7	60	59	−1
Average	42.4	53.6	11.2	79.9	105.5	25.6

The behavior of these heat-treated wheat doughballs was rather unsatisfactory. Many of the doughballs acted in a lifeless manner. More than the usual number of re-checks were required particularly for the high protein series. The high protein Fulhio and Nabob samples gave evidence of receiving some support from the beaker walls. It may, however, be concluded that the heating of these samples did increase the time. It may further be concluded that heat-treated variety samples of wheat are unsuitable for testing by the time test. Unfortunately it was impossible to bake these heat-treated samples due to the small amount of grain available.

Effect of tempering wheat. The amount of moisture in wheat will influence the grinding and meal produced. To obtain some information on the effect of such moisture variations upon time results, a series of 60-g. lots of a high protein (14.0%) Trumbull wheat was exposed to a 90 ± 5% relative humidity in the fermentation cabinet for varying lengths of time. At the end of this exposure the wheat was covered in air-tight bottles and kept until ground. The accumulated samples were ground at the end of 4, 6, and 10 day periods. Moisture determinations and time tests were made upon the freshly ground meals. In making these time tests no additional moisture other than that in the yeast suspension was added. Average results from quadruplicate time tests are given in Table VI.

TABLE VI

EFFECT OF AIR TEMPERING WHEAT
(14.0% protein Trumbull wheat)

Exposed	Moisture in meal	Time	Exposed	Moisture in meal	Time
	%	*Min.*		%	*Min.*
0	11.2	61.3	5 days	14.6	77.0
6 hours	11.7	60.5	6 days	14.8	68.8
14 hours	12.1	62.5	7 days	14.5	81.5
1 day	12.8	63.0	8 days	14.5	82.0
2 days	13.5	61.3	9 days	14.8	86.3
3 days	14.1	68.3	10 days	14.9	89.3
4 days	14.2	73.5			

While the data from these tempering tests are somewhat erratic, yet it is readily observed that a well defined tendency exists for time to increase with length of temper. Chapman and Farquhar (1934) have also observed a higher time from tempered wheat. The " feel " of the doughs varied greatly, the resiliency and smoothness of the dough increasing as the length of temper increased. Apparently most, if not all, of this change was due to the change in moisture content because a sample of Trumbull meal (ground dry) after exposure in the cabinet for 12 hours gave a time of 73 minutes. The moisture content of this tempered meal was 14.7%. These tempering tests indicate the advisability and need for at least reasonably similar moisture contents in the samples which are being time tested. Other tempered samples of the same variety but of varying protein contents were also time tested. These tests indicated that such tempering increased the correlation between time and protein content.

Effect of aging meal. All of the studies reported upon at this time were made upon old, well-matured, sound wheat. Studies dealing with changes in wheat baking strength immediately following harvest

such as reported by Fitz (1910), indicate that time data obtained on newly harvested wheat might easily lead to erroneous conclusions regarding its ultimate strength rating. After the wheat has passed through its period of "sweat" and has thoroughly dried out, the changes in strength are slowed up (Stockham, 1920). The relatively rapid changes occurring in new wheat appear to indicate that the time test should not be used on wheat (if reliable results are desired) until the wheat is six or more weeks old. This impairs its *possible* usefulness to mills situated in sections where a large part of the new annual wheat movement consists of freshly threshed unaged wheat.

Cutler and Worzella (1933) found that ground wheat increased in time more rapidly than in the unground form. To study the effect of age upon meals, two series of samples were used. The series in Table VII were aged only 16 days and the results are very erratic with 6

TABLE VII

EFFECT OF 16 DAYS STORAGE UPON TIME

Variety	Protein (wheat)	Time—4-g. meal		Change due to aging
		Not aged	Aged 16 days	
	%	*Min.*	*Min.*	*Min.*
Trumbull	14.0	67	64	− 3
Nabob	12.8	77	79	+ 2
Fulhio	13.6	64	65	+ 1
Red Rock	13.8	83	90	+ 7
American Banner	12.8	43	47	+ 4
Bald Rock	13.9	67	60	− 7
Michigan Amber	14.0	87	76	−11
Kharkov	14.3	56	50	− 6
Fultz	13.2	81	89	+ 8
Gladden	13.6	54	58	+ 4
Average		67.9	67.8	− 0.1

samples showing a gain, and the remaining 4, a loss, in time due to the aging period. The samples during storage were in air-tight bottles in the laboratory. They were exposed to light but not directly to the sun.

The samples used as the second series were stored under similar conditions to those used in the former. The second series however was kept for 45 days before determining the effect of storage (Table VIII).

These storage tests indicate that to obtain comparable time data, the meals should not be stored very long before the determinations are run. The changes, however, which occur during the first three or four days after grinding are so small that they apparently fall within the experimental error of the test. The changes are not large enough to interfere with the routine of running duplicate determinations on

TABLE VIII

EFFECT OF STORING TWO VARIETIES 45 DAYS

Variety	Moisture (meal)	Protein (wheat)	Time—5-g. meal		Increase due to aging
			Not aged	Aged 45 days	
	%	%	*Min.*	*Min.*	*Min.*
Trumbull	8.9	9.0	44	62	17
"	8.8	10.9	30	36	6
"	8.6	11.2	50	75	25
"	9.7	14.0	52	69	17
Red Rock	8.9	8.8	72	92	20
"	8.7	9.9	50	68	18
"	8.5	10.2	53	75	22
"	9.8	13.8	70	94	24

successive days after grinding. This is the method followed by the author and believed superior to running all tests on a sample at one and the same time.

Conclusions and Summary

A few of the many factors influencing wholemeal time data have been investigated. It is hoped that the presented data may advance knowledge relative to the subject.

It has been shown that the Pelshenke and Cutler-Worzella procedures give definitely different results. With the 150 cc. beaker used as standard equipment at the Ohio Station the author believes a 4-g. doughball will have definite advantages over larger sized doughs provided the entire range in world wheat strength is to be encountered.

The time test is empirical in nature and relative results are the best which may be hoped for. For truly comparable data great care must be taken to see that all tests are made under comparable conditions.

Of the factors studied, variation in vessel diameter proved very important as a cause of variability in the results. The doughball should be of such a size that it receives no support from the vessel used in carrying out the test.

Provided a proper size of vessel is used, the time increases with decreasing size of doughball and increasing moisture in the meal (within the limits studied).

Uniformity in grinding is essential as variations in granulation influence the test.

Samples which have been heated to eliminate insects give time data which are erratic and higher than from the same samples which have not received this heat treatment.

Increases in time due to aging of the meal were found to be within the experimental error of the determination, provided the tests were made within a few days after grinding.

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A SIMPLE METHOD FOR DETERMINING THE "YELLOWNESS" AND "GRADE" OF WHEAT FLOURS

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Introduction

This paper describes a simple method for determining the "yellowness" or the degree of bleaching, in a wheat flour, by determining the amount of carotene pigments present, and its grade, or degree of offal contamination, by determination of the amount of flavones.

No special merit is claimed for it except that it has been found valuable during several years as a routine laboratory method of controlling the colour of flours manufactured by an important group of flour mills.

The method does not pretend to measure the actual colour of flour. This is a problem of very considerable difficulty, and although the recent work of Baker *et al.*[1] represents an enormous advance towards its solution, there is as yet no method for the direct optical determination of the colour of flour which can be left in the hands of any except skilled workers.

The bibliography[2] of the occurrence and measurement of carotenes in wheat and its products is copious; there is no need for any reference to it here.

It is only quite recently that references to the presence of flavones in flour have been made.[3] The writer, whose acquaintance with cereal chemistry commenced in 1906, early noticed that flour gave a more or less strong yellow colour with alkalies, which he thought, from the wide-spread occurrence of flavones in plant life, might be due to the presence of flavone-like pigments. The practical interest of this observation was that it gave an explanation of the long-known fact that self-raising flours containing an excess of sodium bicarbonate over the acid ingredient, developed an objectionable yellow or brownish colour in the crumb of the baked goods.

In 1926, in the course of an investigation into the pigments of wheat, this question of the occurrence of flavones again came to the

[1] Baker, J. C., Parker, H. K., and Freese, F. B. The measurement of color in flour and bread by means of Maxwell discs. Cereal Chem. **10**: 437–446 (1933).

[2] Geddes, W. F., Binnington, D. S., and Whiteside, A. G. O. A simplified method for the determination of carotene in flour extracts. Cereal Chem. **11**: 1–24 (1934).

[3] Markley, M. C., and Bailey, C. H. The pigments of the dilute alcohol or acetone extract of whole wheat meal. Cereal Chem. **12**: 40–48 (1935).

fore. It was observed that high-grade flours gave only a weak yellow colour with alkalies, lower-grade flours a stronger colour, germ an intense yellow, and that the brown colour of bran from red wheats became a strong orange-brown colour, while bran from white wheats turned an intense yellow. This indicated that flavones were much more strongly concentrated in the envelope of the wheat grain than in the endosperm.

It was further found that the carotene pigments could be easily and practically completely extracted from flour by cold (100%) acetone, and that then from the same portion of flour, flavones could then be extracted with 80% acetone, the addition of ammonia to the latter giving a perfectly clear yellow solution, whose colour depended on the grade of the flour, the depth of the yellow colour being measurable in any of the usual types of colorimeter. The Klett Biocolorimeter was actually used in this estimation.

Method

The method finally adopted is as follows:

Into the stem of an 8 cm. conical funnel is rather tightly pushed a plug of filter paper, so as to be about one-eighth of an inch thick; over this a plug of cotton wool is placed, packed tightly over the filter paper plug, but extending up into the conical part of the funnel to a distance of about half-an-inch. The funnel is fitted into the neck of a 100 cc. filter flask with a rubber stopper, the side tube of the flask being connected to a vacuum pump. A dozen or so such units may be connected up to the vacuum pump by making a pipe line of glass T tubes, connecting each flask to a T tube with a glass stop-cock, or pressure tube and pinch-cock.

5 g. of flour is put into the funnel, and for the determination of the yellow carotene colour, 50 cc. of 100% acetone is added in successive portions of about 10 cc., the stop-cock being so adjusted that the 50 cc. of acetone run through in about 15 minutes. As each 10 cc. portion of acetone is added, the flour-acetone mixture is well stirred with a short glass rod which remains in the funnel. On completion of the extraction, the carotene solution is at once transferred to the appropriate tube of the colorimeter, and its colour compared with that of an acidified 0.01% solution of potassium chromate in water. In taking the readings, it is found advisable to obtain the comparison between the colours of the unknown and standard solutions by first lowering the plunger (in the Klett apparatus) in the standard tube so that the colour at first appears deeper than that of the unknown, taking the reading when the comparison appears to be exact, then depressing the plunger still further and gradually raising it until the comparison again appears exact. This operation should be repeated, the final figure taken being the average of these four readings.

The flavones are then immediately extracted with 50 cc. of 80% acetone (80 cc. acetone made up to 100 cc. with water) in exactly the same way. Distinct flocculation of the flour occurs in 80% acetone, so that filtration is slower. Filtration should be completed in about one hour. A perfectly bright extract is obtained which remains perfectly bright on adding 2 cc. of strong (Sp. Gr. 0.880) ammonia. The yellow extract is then poured into the colorimeter tube. In both cases the final volume of the extract is made up to exactly 50 cc., washing out the flask with a little 100% or 80% acetone as the case may be. The yellow colour is compared with that of a 0.01% solution of K_2CrO_4 having a pH of 6.2.

With ordinary care, no difficulty should be found in fitting in the filter paper and cotton wool plugs with the requisite degree of tightness, although at first a little experience may be necessary. The chief object of the paper plug is to prevent the passage of cotton wool fibre into the flask. It is unnecessary to ram it in very tightly. The cotton-wool plug must fit tightly over the paper plug and must extend quite half-an-inch up into the conical part of the funnel. The plugs must be adjusted

so that perfectly bright extracts are obtained, and at the same time filtration does not take longer than the times indicated. Only gentle suction should be necessary.

The extraction with 80% acetone must follow that with 100% acetone as quickly as possible, or the flour becomes lumpy, the extraction difficult, and the extracts may be turbid.

The use of specially purified acetone is unnecessary. The cheap spirit known in England as "redistilled acetone" is sufficiently pure. It was found that 80% acetone gave the maximum development of yellow colour with a perfectly clear extract, stronger acetone gave weaker colours and weaker acetone filtered badly and gave turbid extracts.

To prevent evaporation of the acetone, the funnel should be covered with a glass plate, and in hot weather it may be necessary to use, instead of the conical funnel, a cylindrical filter tube of about the same capacity, with a loosely-fitting rubber stopper.

For use with the colorimeter a daylight lamp should be obtained, with a reasonably constant line voltage. A simple form of hood to exclude extraneous light should be provided. Otherwise, no special precautions are necessary.

It is not claimed that the extraction as described with 80% acetone removes all flavones. For instance, allowing the flour, after carotene extraction, to stand overnight in 80% acetone, gives a distinctly higher "flavone yellow" figure. Probably several flavones are present in wheat in different kinds of combination as glucosides, etc., and having different degrees of solubility. But the method gives concordant results on the same sample as can be seen by examining Table I. On

TABLE I

RESULTS OF NINE INDEPENDENT DETERMINATIONS ON THE SAME FLOUR

All the figures quoted refer to the depth in millimetres of the standard potassium chromate solutions required to match the colour of a 60 mm. column of the acetone extracts.

Test number	Carotene colour	Flavone colour
1	12.0	21.0
2	11.0	20.0
3	13.0	20.5
4	12.0	20.5
5	13.0	19.5
6	12.0	22.0
7	12.0	21.0
8	11.5	20.5
9	12.0	19.5
Average	12.3	20.5

Tests on five flours, carried out on one day and repeated the next day.

Flour number	Carotene colour		Flavone colour	
	1st day	2d day	1st day	2d day
1	12.30	14.00	7.75	8.25
2	16.00	15.00	8.75	8.75
3	9.25	9.75	15.00	14.25
4	19.00	19.50	13.50	13.25
5	10.25	11.00	17.75	17.00

the other hand, the extraction with pure acetone does seem to remove all carotenes, bearing in mind that the method is only advocated as a routine commercial method.

Recovery of the Acetone

100% acetone: The carotene extracts are put into a stock bottle containing pieces of fused calcium chloride, and after standing at least 24 hours, the supernatant acetone layer is removed and distilled off.

80% acetone: The flavone extracts are transferred to another stock bottle, and when a sufficient quantity has accumulated, the amount of dilute sulphuric acid required to neutralise an aliquot portion, diluted with an equal volume of water, is determined by titration, using methyl orange as indicator. The whole bulk is then neutralised with the same acid.

After standing a few hours, a strong watery solution of ammonium sulphate, containing much of the water present, separates out, the top acetone layer is removed and distilled off until the temperature of the vapour, after remaining steady at about 70° C., suddenly begins to rise.

This yields acetone of approximately 90% strength, which is then diluted to 80% using the hydrometer. 80% acetone has a Sp. Gr. of 0.865.

Care should be taken that the acidity of the 80% acetone is not too high, otherwise turbid extracts may result. One drop (0.05 cc.) of 0.1 N. NaOH added to 20 cc. 80% acetone, should give a permanent pink colour using phenol red indicator.

Standard Potassium Chromate Solutions

(1) For matching carotene colours:

20 cc. of a 0.1% potassium chromate solution is made up to 200 cc. including 2 cc. 0.1 N. H_2SO_4. This solution is 0.01% K_2CrO_4 almost completely converted to dichromate.

(2) For matching flavone colours:

Dilute 20 cc. of the 0.1% potassium chromate stock solution to 200 cc. using Clark's phosphate buffer solution of pH 6.2 (add 8.60 cc. 0.2 N. NaOH to 50 cc. 0.2 M. KH_2PO_4, dilute to 200 cc.).[4]

Advantages of the Method

It is simple, requires no more elaborate apparatus than the colorimeter, and can be worked by an unskilled operative, with a little training.

It is rapid, and as regards the flavone determination at least as accurate as and more sensitive than an ash determination, besides being less troublesome. (See Table II.) Twelve samples can easily

TABLE II

COMPARISON BETWEEN FLAVONE FIGURES AND ASH CONTENTS OF DIFFERENT GRADES OF FLOUR

The figures in each case represent the averages of many routine determinations.

Grade of flour	Flavone colour		Ash content	
	Figure	Rating	%	Rating
Top Patents	9	= 1.00[1]	0.35	= 1.00[1]
1/– Patents	14	= 1.56	0.42	= 1.20
Standard	16	= 1.78	0.45	= 1.25
Households	20	= 2.22	0.50	= 1.43
Specials	24	= 2.67	0.55	= 1.57

[1] For more ready comparison, the figures for Top Patents are taken as unity in each case.

be tested in a working day by one operator.

It is economical, requiring only 5 g. of flour, and only *one* solvent is used. The residues are easily treated so as to obtain a high recovery of acetone.

[4] Jørgensen, Holger. Use of chromate-solutions as comparison-standards for the determination of "gasoline colour values." Cereal Chem. 4: 468–469 (1927).

In view of the high "flavone-yellow" figures for bran, and especially for germ, it is valuable as giving approximate data as to the destination of bran and germ in the flour-milling system. This figure is of much greater value than the ash content. This is brought out by the data shown in Table III.

TABLE III

RELATIVE VALUE OF FLAVONE AND ASH CONTENT FIGURES

Material	Flavone colour		Ash content	
	Figure	Rating	%	Rating
Top Patents	9 =	1	0.35 =	1
Specials	24 =	2.67	0.55 =	1.57
Bran	600 =	66	6.00 =	17.0
Germ	1600 =	177	4.50 =	13.0

In English mills at any rate, it is not usually possible to extract more than 0.5% of the wheat ground in the form of reasonably pure germ, whereas wheat is generally said to contain about 2% of germ. An investigation in one particular mill indicated that the residual germ found its way almost entirely into the middlings, which contained about 17% of germ, a result which emphasizes the value of wheat-middlings as a feedstuff, in view of the high vitamin activity of wheat germ.

It is necessary to say *approximate* data in this connection, since the flavone yellow of bran is a brownish-yellow, and that of germ a greenish-yellow, which only compare moderately closely with that of the standard chromate colour.

The value of the method as an aid in the control of the flour mill will be obvious. It provides a simple means of regularising the degree of bleach and the grade of the manufactured flours. Table IV illustrates this point.

TABLE IV

EFFECT OF BLEACHING ON CAROTENE COLOUR

Grade of flour	Flour treatment	Carotene figure
Top Patents	unbleached	20
" "	bleached (Exothermic)	16
Specials	unbleached	23
"	bleached (Beta Chlora)	16
Specials	unbleached	19
"	bleached (Agene)	7
Top Patents	bleached (Exothermic)	10
" "	bleached (Exothermic and Novadel)	2

It is useful for determining the grade of flours which have been treated with "mineral" improvers, or of self-raising flours, where an ash determination cannot be applied.

The method does not pretend to be one for the determination of the real colour of flour; of flours of equal grade from red and white wheats respectively, that from white wheat will always appear to be clearer than that from red, while the evidence is that the "flavone yellow" colour from white bran is at any rate nearly as intense as that from red wheat. This may in fact be turned to account when it is required to ascertain the type of wheat from which an "unknown" flour has been made. The data given in Table V illustrates this point.

TABLE V

Information was required as to the character of four competing flours, all sold as Top Patents in the same area. "B" appeared a trifle clearer, but yellower than "A," in the Pekar test and in the crumb colour of the bread. "C" was of a much stronger yellow colour, but still quite clear. "D" was dingy, and much inferior to the others in colour.[1, 2]

Flour number	Carotene figure	Flavone figure
A	15.5	8.5
B	20.5	7.0
C	32.5	15.3
D	16.0	17.5

[1] "A" and "D" were evidently just moderately bleached, "B" probably unbleached, and "C" unbleached with a remarkably high yellow colour.

[2] "B" was the highest in grade, "A" rather lower; "C" was obviously not, as regards grade, to be classed as a Top Patent, and was evidently made from white wheats, while "D" again was not a Patent flour at all, being of quite low grade.

Summary

A simple, rapid method for determining the "yellowness" and grade of a flour is described.

No more elaborate apparatus than a standard type of colorimeter is required, the rest of the equipment being ordinary laboratory glassware.

The method depends on the extraction of flour with pure acetone, from which the yellow colour due to carotenes can be determined, and again with 80% acetone, from which, after making alkaline, measurement of the "flavone yellow" colour gives a measure of the grade of the flour.

As regards the determination of the "grade" of a flour, the flavone colour determination gives a more sensitive method than ash content determination, and is applicable where the latter cannot be used.

Acknowledgment

I wish to acknowledge the valuable services rendered by a former assistant, Mr. F. R. Charles, in working out the details of the method.

REPORT OF THE COMMITTEE ON STANDARDIZATION OF METHODS FOR BREWING AND MALTING CONTROL

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Bureau of Agricultural Economics, U. S. Department of Agriculture, Washington, D. C.

(Read at the Annual Meeting, June 1935)

At the Toronto Meeting of the A. A. C. C. the incoming president appointed a committee whose activities were to undertake the standardization of methods of analysis for brewing and malting control.

The members of this committee were D. A. Coleman, Chairman, H. C. Gore, H. D. Rohde, James D. Veron, Elsie Singruen, and Donald Williams.

The committee held its first meeting at Chicago in September 1934, and discussed with interested malt and brewery chemists, the need of standard methods for the analysis of malt. From the discussions which took place it was very evident that there was need for standardization in this branch of cereal chemistry and that something should be done as soon as practical with respect to the standardization of methods for the analysis of malt and malt adjuncts.

At the first meeting it was agreed to sponsor as a tentative official group of methods of analysis for malt, the methods described in the Salzburg Agreement. Through the generosity of Miss Singruen a translation of these methods was made from the German and furnished to all interested parties.

In the mean while, in order that the committee might have some first hand facts and figures relative to malt analysis to use as a guide in future activities, two samples of malt were sent to more than thirty different laboratories who engage in the analysis of malt, with the request that they analyze these samples by their own individual methods, and for such factors as they considered important in evaluating the quality of malt.

Each analyst was asked, in submitting his analytical results, to furnish the chairman of this committee with a statement of the methods of analysis used by him, so that this information would be available for use in interpreting such analytical variances which might occur.

It was expected, with the consummation of the above plan of

TABLE I

RESULTS OF PHYSICAL AND CHEMICAL DETERMINATIONS MADE BY 18 COLLABORATORS ON SAMPLE OF MALT (NO. 1 UNGROUND) SENT OUT FROM WASHINGTON, D. C., NOVEMBER 15, 1934

Determinations	Collaborator number						
	1	2	3	4	5	6	7
Physical examination							
Acrospire growth							
0–1/4, %	6.5	3	—	6	—	5	—
1/4–1/2, %	1.0	3	7	1	—	1	—
1/2–3/4, %	5.5	4	16	19	—	5	—
3/4–1, %	80.5	88	77	68	—	81.5	—
Over 1, %	3.0	2	—	6	—	7.5	—
Assortment	—	—	—	—	—	—	—
Foreign seeds, %	1.0	—	—	—	—	.0	—
Mellow, %	97.5	91.3	—	94.0	—	84	—
Dark brown kernels, %	—	—	—	—	—	—	—
Light brown kernels, %	—	—	—	—	—	—	—
Half-glassy kernels, %	—	—	—	3.0	—	10	—
Hard kernels, %	—	—	—	—	—	—	—
Intermediate hard kernels, %	—	—	—	—	—	—	—
Part-glassy kernels, %	2.0	—	—	—	—	—	—
White and mealy kernels, %	—	—	—	—	—	—	—
White and hard kernels, %	—	—	—	—	—	—	—
Wholly-glassy kernels, %	1.5	—	—	3.0	—	6	—
Yellow and hard kernels, %	—	—	—	—	—	—	—
Yellow and soft kernels, %	—	—	—	—	—	—	—
Mold	—	—	—	—	—	None	—
Sizing tests							
% on top of 1/2 × 8/64″ sieve	—	—	—	—	—	33.0	—
% on top of 1/2 × 7/64″ sieve	22.4	—	—	—	—	32.2	—
% on top of 1/2 × 6¼/64″ sieve	44.5	—	—	—	—	28.6	—
% on top of 1/2 × 5½/64″ sieve	31.5	—	—	—	—	5.8	—
% on top of 1/2 × 5/64″ sieve	—	—	—	—	—	.2	—
% on top of 1/2 × 4½/64″ sieve	.7	—	—	—	—	.2	—
% passing through 1/2 × 4¼/64″ sieve	.1	—	—	—	—	—	—
Weight per bushel, *lbs.*	—	40.3	39.0	—	—	39.1	—
Weight per 1,000 kernels, *gms.*	30.09	—	—	30.00	—	—	—
Chemical determinations							
Malt—							
Moisture content, %	5.69	4.95	6.60	5.40	5.46	5.40	5.15
Acidity, % as lactic	—	.756	—	—	—	—	—
Ash content, %	—	2.31	—	—	—	—	—
Diastatic activity, °Lintner,	125	129	84	85	—	120	120
Extract, fine grist, as is basis, %	69.96	68.19	—	68.40	67.80	69.05	70.01
(*a*) Extract, fine grist, dry basis, %	74.18	71.74	70.60	72.30	71.70	73.00	73.80
Extract, coarse grind, as is basis, %	68.26	—	—	67.30	—	—	—
(*b*) Extract, coarse grind, dry basis, %	72.38	—	—	71.30	—	—	—
Difference, *a–b*	1.80	—	—	1.00	—	—	—
Protein, dry basis, %	15.86	—	15.54	—	—	—	—
Wort—							
Acidity, % as lactic	—	—	—	—	—	—	—
Appearance of wort	Hazy	Clear	—	Brilliant	—	Clear	—
Color of wort	—	—	—	—	44 S *c/*	—	—
Color of wort, using Lovibond ¼″ cell and Series 52 glasses	1.50	1.65	—	1.60	—	1.7	—
Filtration of wort	Rapid	Rapid	—	—	Rapid	—	—
Odor of wort	—	—	—	—	—	—	—
Specific gravity—							
at 15° C.	—	—	1.03440	1.03150	—	—	—
at 17.5° C.	—	*d/*	—	—	—	—	—
at 20° C.	—	—	—	—	1.03091	1.03140	1.03097
Time of conversion at 70° C., *minutes*	5	5	—	5	—	5	—
Tables used for converting to per cent extract and/or yield	Spencer-Meade Table 31	Doemens	Balling-Wahl-Henius	Balling-Windisch	Plato	Meindl-Plato	Windisch

a/ Non-buffered.
b/ Buffered.
c/ Stamner units.
d/ Made at 17.5° C. but not reported.

TABLE I—*Continued*

Collaborator number											Average
8	9	10	11	12	13	14	15	16	17	18	
5	1	3	—	3	6	—	10	2	—	1	4.3
0	3	3	—	3	3	—	0	0	—	5	2.3
1	4	1	—	4	4	—	13	3	—	7	6.6
91	90	93	—	87	85	—	74	89	—	87	83.9
3	2	0	—	3	2	—	1	6	—	0	3.0
—	Satisfactory	—	—	—	—	—	—	—	—	—	—
.0	.5	.0	—	.0	—	—	—	—	—	—	—
Good	95	90 V.G.	—	—	92	—	74	—	—	—	—
—	—	—	—	—	—	1	—	—	—	—	—
—	—	—	—	—	—	1	—	—	—	—	—
—	—	—	—	2	—	—	—	—	—	—	—
—	—	—	—	—	—	—	17	—	—	—	—
—	—	—	—	—	—	—	9	—	—	—	—
—	—	—	—	—	—	—	—	—	—	—	—
—	—	—	—	90	—	94	—	90	—	—	—
—	—	—	—	4	—	3	—	8	—	—	—
—	—	—	—	2	—	0	—	1	—	—	—
—	—	—	—	0	—	—	—	—	—	—	—
—	—	—	—	2	—	—	—	1	—	1	—
None	None	None	—	None	—	—	—	—	—	—	None
—	—	—	—	—	—	—	—	—	—	—	—
—	—	—	—	—	—	—	—	—	—	—	—
—	—	—	—	—	87.0	—	—	—	—	—	—
—	—	—	—	—	10.1	—	—	—	—	—	—
—	—	—	—	—	2.2	—	—	—	—	—	—
—	—	—	—	—	.7	—	—	—	—	—	—
—	—	—	—	—	—	—	—	—	—	—	—
39.0	39.0	38.5	—	39.7	39.5	—	39.4	—	—	39.0	39.25
—	—	—	—	—	—	28.80	29.36	—	—	—	29.56
5.00	5.29	5.27	4.32	5.70	5.45	4.92	5.98	5.40	5.93	4.90	5 38
—	—	—	—	—	—	—	—	—	—	—	.756
—	—	—	—	—	—	—	—	—	—	—	2.31
150	—	116	153	132	129	—	139 *a*/ 150 *b*/	117	115	—	123
68.79	68.46	70.23	64.27	70.46	68.66	69.33	69.01	70.31	—	69.31	68.89
72.41	72.29	74.12	67.17	74.70	72.66	72.92	73.38	74.33	—	72.88	72.60
—	—	68.13	—	—	67.09	—	—	68.81	—	—	67.91
—	—	71.91	—	—	70.99	—	—	72.73	—	—	71.86
—	—	2.10	—	—	1.67	—	—	1.60	—	—	1.63
—	—	—	—	—	—	—	15.35	—	—	—	15.58
—	.11	—	.99	—	—	—	—	—	—	—	—
—	Brilliant	Brilliant	—	Clear	Brilliant	Normal	Normal	Clear	—	Clear	Clear
—	Very pale straw	Pale straw	46 *s*	—	—	—	—	—	—	—	—
1.87	1.50	1.90	—	1.50	1.70	—	—	1.75	—	1.50	1.65
—	Fast	Rapid	—	Rapid	Fast	Normal	Rapid	—	—	Rapid	Rapid
—	Aromatic	—	—	—	—	—	—	—	—	—	—
—	—	—	—	1.03150	—	—	—	1.03144	—	—	1.03221
—	—	*d*/	—	—	—	—	—	—	—	—	
1.03130	—	—	—	—	1.03089	1.03143	1.03135	—	—	—	1.03118
5	5	5	—	5	5	Under 10	10–15	5	—	5	5
Meindl-Plato	—	Schultz-Ostermann	—	Schultz-Ostermann	Plato	A.O.A.C. sugar table	Plato-Pawlowski	Schultz-Ostermann	—	—	—

TABLE II

RESULTS OF CHEMICAL TESTS MADE BY 15 COLLABORATORS ON ABOVE SAMPLE OF MALT (SAMPLE NO. 1) BUT GROUND AT WASHINGTON, D. C., BEFORE SENDING TO VARIOUS COLLABORATORS

Chemical determinations	Collaborator number						
	1	2	3	4	5	6	7
Malt—							
Moisture content, %	5.65	4.85	6.50	5.10			5.57
Acidity, % as lactic	—	.756	—	—			—
Ash content, %	—	—	—	—			—
Diastatic activity, °Lintner,	122	129	85	95			114 *a/* 133 *b/*
Extract, fine grist, as is basis, %	69.39	68.14	—	68.40			70.05
Extract, fine grist, dry basis, %	73.54	71.62	72.49	72.10			74.18
Protein content, dry basis, %	15.02	—	15.72	—			—
Wort—							
Acidity, % as lactic	—	—	—	—			—
Appearance of wort	Clear	Clear	—	Brilliant			—
Color of wort	—	—	—	—			—
Color of wort, using Lovibond 1" cell and Series 52 glasses	1.5	1.7	—	1.6			—
Filtration of wort	Rapid	Rapid	—	Fast			—
Odor of wort	—	—	—	—			
Specific gravity—							
at 15° C.	—	—	1.0353	1.03150			—
at 17.5° C.	—	*d/*	—	—			—
at 20° C.	—	—	—	—			1.03107
Time of conversion at 70° C., *minutes*	5	5	—	5			—
Tables used for converting to per cent extract and/or yield	Spencer-Meade Table 31	Doemens	Balling-Wahl-Henius	Balling-Windisch	Sample not analyzed by collaborator	No sample sent to collaborator	Windisch

a/ Non-buffered.
b/ Buffered.
c/ Stamner units.
d/ Made at 17.5° C. but not reported.

study, that information would develop with respect to uniformity in the reporting of analytical data as well as furnish information with regard to the magnitude of analytical variances to be expected at this time between different malt analysts. With such information available, plans for the standardization of analytical procedures for malt should more logically evolve.

Material for Study

Two samples of malt, one of low diastatic activity and the other of high diastatic activity, were made available to the committee through the courtesy of the Fleischmann Company.

Preparation of Samples

To insure as complete uniformity in the composition of the various lots of malt sent to each analyst as possible, two bushel lots of the malt were thoroughly mixed by passing through a Boerner sampler (see U. S. Dept. Agr. Bull. 1065) fifty times. A quart sample of each lot of unground malt was sent to each analyst. Similarly, ground samples of the same two malts were prepared by grinding in a Labconco

TABLE II—*Continued*

Collaborator number											Average
8	9	10	11	12	13	14	15	16	17	18	
5.30	5.29	5.11	3.95	5.60	5.35	No sample sent to collaborator	5.45	5.40	5.78	5.15	5.40
—	—	—	—	—	—		—	—	—	—	—
—	—	—	—	—	—		—	—	—	—	—
148	—	122	153	134	129		—	117	117	—	124
69.28	68.46	69.81	64.24	70.01	68.14		69.06	70.41	—	69.82	68.86
73.16	72.29	73.58	66.88	74.11	72.03		73.00	74.43	—	73.61	72.64
—	—	—	—	—	—		15.29	—	—	—	15.34
—	.11	—	.99	—	—		—	—	—	—	.55
Brilliant	Brilliant Very pale straw	Brilliant Pale straw	— 46 *c*/	Clear	Brilliant		—	Clear	—	Clear	Clear
—	1.5	1.9	—	1.5	1.8		—	1.75	—	1.50	1.66
1.87 Rapid	Fast Aromatic	Rapid	—	Rapid	Fast		Rapid	—	—	Rapid	Rapid
—	—	—	—	1.03124	—		—	1.03148	—	—	1.03238
—	—	*d*/	—	—	—		—	—	—	—	—
1.03130	—	—	—	—	1.03101		1.03140	—	—	—	1.03119
5	5	5	—	5	5		10–15	5	—	5	5
Meindl-Plato	—	Schultz-Ostermann	—	Schultz-Ostermann	Plato		Plato-Pawlowski	Schultz-Ostermann	—	—	—

grinder set to grind at XF. A Seck mill was not available. While it is admitted the results of the tests made on the samples ground in the Labconco grinder might not correlate exactly with results obtained on grist pulverized by the Seck mill, due to differences in the fineness of division of the sample, the moisture results on the ground sample offer an opportunity to study and to determine to what extent preparation of the sample has on analytical variances.

Analytical Results

Analytical data on these four lots of malt were furnished by 18 of the 30 analysts to whom the samples were sent, and are given in Tables I, II, III, and IV. The analytical data submitted divide themselves clearly into two groups—physical and chemical.

Physical Determinations

The physical determinations made by the different collaborators were, acrospire growth, assortment, foreign seeds, mellowness, presence of mold, sizing tests, weight per bushel, and weight per 1,000 kernels. Acrospire growth, mellowness of malt, and weight per bushel appear to be the three outstanding factors of quality reported by the 18 collaborators. However, only 11 of the 18 collaborators reported on

TABLE III

RESULTS OF PHYSICAL AND CHEMICAL DETERMINATIONS MADE BY 18 COLLABORATORS ON SAMPLE OF MALT (NO. 2 UNGROUND) SENT OUT FROM WASHINGTON, D. C., NOVEMBER 15, 1934

Determinations	Collaborator number						
	1	2	3	4	5	6	7
Physical examination							
Acrospire growth							
0–1/4, %	2.5	2	—	5	—	2	—
1/4–1/2, %	1.5	2	6	4	—	0	—
1/2–3/4, %	6.5	14	18	31	—	4.5	—
3/4–1, %	89.0	78	76	60	—	85	—
Over 1, %	.5	4	—	0	—	8.5	—
Assortment	—	—	—	—	—	—	—
Foreign seeds, %	2.0	—	—	—	—	Oats, wheat	—
Mellow, %	—	92.52	—	—	—	88.5	—
Dark brown kernels, %	—	—	—	—	—	—	—
Light brown kernels, %	—	—	—	—	—	—	—
Half-glassy kernels, %	—	—	—	8	—	10	—
Hard kernels, %	—	—	—	—	—	—	—
Intermediate hard kernels, %	—	—	—	—	—	—	—
Part-glassy kernels, %	5.0	—	—	—	—	—	—
White and mealy kernels, %	90.0	—	—	88	—	—	—
White and hard kernels, %	—	—	—	—	—	—	—
Wholly-glassy kernels, %	5.0	—	—	4	—	8	—
Yellow and hard kernels, %	—	—	—	—	—	—	—
Yellow and soft kernels, %	—	—	—	—	—	—	—
Mold	—	—	—	—	—	None	—
Sizing tests							
% on top of 1/2 × 8/64″ sieve	—	—	—	—	—	5.4	—
% on top of 1/2 × 7/64″ sieve	1.0	—	—	—	—	8.4	—
% on top of 1/2 × 6½/64″ sieve	9.1	—	—	—	—	41.8	—
% on top of 1/2 × 5½/64″ sieve	—	—	—	—	—	33.2	—
% on top of 1/2 × 5/64″ sieve	74.8	—	—	—	—	6.4	—
% on top of 1/2 × 4½/64″ sieve	13.5	—	—	—	—	4.8	—
Passing through 1/2 × 4½/64″ sieve, %	1.0	—	—	—	—	—	—
Weight per bushel, *lbs.*	—	39.0	40.0	—	—	39.8	—
Weight per 1,000 kernels, *gms.*	—	—	—	22.4	—	—	—
Chemical determinations							
Malt—							
Moisture content, %	6.58	5.95	6.90	6.40	6.58	6.20	6.68
Acidity, % as lactic	—	.738	—	—	—	—	—
Ash content, %	—	—	—	—	—	—	—
Diastatic activity, °Lintner,	208	180	145 *a*/	150 *a*/	—	175	150 *a*/ 160 *b*/
Extract, fine grist, as is basis, %	67.29	65.87	—	65.90	66.20	66.56	67.30
(*a*) Extract, fine grist, dry basis, %	72.03	70.03	69.53	70.50	70.90	70.96	72.10
Extract, coarse grind, as is basis, %	64.64	—	—	64.30	—	—	—
(*b*) Extract, coarse grind, dry basis, %	69.19	—	—	68.80	—	—	—
Difference, *a*–*b*	2.84	—	—	1.70	—	—	—
Protein, dry basis, %	16.39	—	16.31	—	—	—	—
Wort—							
Acidity, % as lactic	—	—	—	—	—	—	—
Appearance of wort	Hazy	Clear	—	Clear	—	Clear	—
Color of wort	—	—	—	—	42 S *c*/	—	—
Color of wort, using Lovibond ¼″ cell and Series 52 glasses	1.50	1.70	—	1.60	—	1.50	—
Filtration of wort	Rapid	Rapid	—	Fast	—	Rapid	—
Odor of wort	—	—	—	—	—	—	—
Specific gravity—							
at 15° C.	—	—	1.0338	1.0304	—	—	—
at 17.5° C.	—	*d*/	—	—	—	—	—
at 20° C.	—	—	—	—	1.0301	1.0314	1.03019
Time of conversion at 70° C., *minutes*	5	5	—	5	—	5	—
Tables used for converting to per cent extract and/or yield	Spencer-Meade Table 31	Doemens	Balling-Wahl-Henius	Balling-Windisch	Plato	Meindl-Plato	Windisch

a/ Non-buffered.
b/ Buffered.
c/ Stamner units.
d/ Made at 17.5° C. but not reported.

TABLE III—*Continued*

Collaborator number											Average
8	9	10	11	12	13	14	15	16	17	18	
1	1	4	—	6	4	—	2	2	—	1	2.7
0	2	2	—	1	3	—	5	1	—	4	2.4
0	8	4	—	5	6	—	9	2	—	9	9.0
95	89	90	—	88	86	—	74	95	—	86	83.9
4	0	0	—	0	1	—	10	0	—	0	2.3
—	OK	—	—	—	—	—	—	—	—	—	—
None	1¼	None	—	—	—	—	—	—	—	—	—
Good	95	90	—	—	90	—	—	—	—	—	—
—	—	—	—	—	—	—	—	—	—	—	—
—	—	—	—	—	—	2	—	—	—	—	—
—	—	—	—	—	—	2	—	—	—	—	—
—	—	—	—	4	—	—	14	1	—	—	—
—	—	—	—	—	—	—	18	—	—	—	—
—	—	—	—	—	—	—	—	—	—	—	—
—	—	—	—	83	—	92	68	90	—	—	—
—	—	—	—	5	—	4	—	8	—	—	—
—	—	—	—	7	—	—	—	—	—	—	—
—	—	—	—	1	—	—	—	—	—	—	—
—	—	—	—	0	—	—	—	—	—	—	—
None	None	None	—	None	—	—	—	—	—	—	None
—	—	—	—	—	—	—	—	—	—	—	—
—	—	—	—	—	—	—	—	—	—	—	—
—	—	—	—	—	35.2	—	—	—	—	—	—
—	—	—	—	—	38.0	—	—	—	—	—	—
—	—	—	—	—	17.5	—	—	—	—	—	—
—	—	—	—	—	9.4	—	—	—	—	—	—
—	—	—	—	—	—	—	—	—	—	—	—
40.0	40.0	39.0	—	40.5	40.0	—	40.4	39.4	—	40.5	39.87
—	—	—	—	—	—	23.6	22.6	—	—	—	22.8
6.00	6.10	5.76	5.16	6.60	6.33	6.24	6.69	6.20	6.73	5.65	6.26
—	—	—	1.03	—	—	—	—	—	—	—	.884
—	—	—	—	—	—	—	2.34	—	—	—	—
185	—	179	189	193	185	—	171	167	164 a/	—	175
65.86	66.17	67.80	65.62	67.79	66.46	67.25	66.14	68.40	—	67.30	66.74
70.06	70.47	71.94	69.19	72.57	70.93	71.72	70.88	72.91	—	71.33	71.06
—	—	65.05	—	—	65.05	—	—	65.94	—	—	65.00
—	—	69.03	—	—	69.43	—	—	70.29	—	—	69.39
—	—	2.91	—	—	1.50	—	—	1.62	—	—	2.11
—	—	—	—	—	—	—	16.77	—	—	—	16.49
—	.11	—	—	—	—	—	—	—	—	—	—
Brilliant	Brilliant	Brilliant	—	Clear	Brilliant	—	—	Clear	—	Clear	Clear
—	Very pale straw	Pale straw	43 s	—	—	16–18 cc. N/10 I.	—	—	—	—	—
—	1.60	1.90	—	1.60	1.80	—	—	1.75	—	1.20	1.62
—	Rapid	Rapid	—	Rapid	Fast	Normal	Rapid	—	—	Rapid	Rapid
—	—	—	—	—	—	Normal	—	—	—	—	—
—	—	—	—	1.03022	—	—	—	1.03056	—	—	1.03124
—	—	d/	—	—	—	—	—	—	—	—	—
1.0300	—	—	—	—	1.02994	1.03052	1.0301	—	—	—	1.03072
5	5	5	—	5	5	Under 10	5–10	—	—	5	5
Meindl-Plato	—	Schultz-Ostermann	—	Schultz-Ostermann	Plato	A.O.A.C. sugar table	Plato-Pawlowski	Schultz-Ostermann	—	—	—

TABLE IV

RESULTS OF CHEMICAL TESTS MADE BY 15 COLLABORATORS ON ABOVE SAMPLE OF MALT (SAMPLE NO. 2) BUT GROUND AT WASHINGTON, D. C., BEFORE SENDING TO VARIOUS COLLABORATORS

Chemical determinations	Collaborator number						
	1	2	3	4	5	6	7
Malt—							
Moisture content, %	6.65	5.85	7.70	6.00	—	—	6.62
Acidity, % as lactic	—	.756	—	—			—
Ash content, %	—	—	—	—			—
Diastatic activity, °Lintner,	208	180	139 a/	165 a/			150 a/ 163 b/
Extract, fine grist, as is basis, %	67.96	65.77	—	65.90			67.30
Extract, fine grist, dry basis, %	72.80	69.86	71.35	70.20			72.05
Protein content, dry basis, %	16.31	—	16.31	—			—
Wort—							
Acidity, % as lactic	—	—	—	—			—
Appearance	Clear	Clear	—	Clear			—
Color of wort	—	—	—	—			—
Color of wort, using Lovibond ½" cell and Series 52 glasses	1.50	1.60	—	1.60			—
Filtration of wort	Rapid	Rapid	—	Fast			—
Odor of wort	—	—	—	—			—
Specific gravity—							
at 15° C.	—	—	1.0343	1.0304			—
at 17.5° C.	—	d/	—	—			—
at 20° C.	—	—	—	—			1.03029
Time of conversion at 70° C., *minutes*	5	5	—	5			—
Tables used for converting to per cent extract and/or yield	Spencer-Meade Table 31	Doemens	Balling-Wahl-Henius	Balling-Windisch	Sample not analyzed by collaborator	No sample sent to collaborator	Windisch

a/ Non-buffered.
b/ Buffered.
c/ Stamner units.
d/ Made at 17.5° C. but not reported.

acrospire growth, only 10 reported on bushel weight, while only 9 reported on mellowness of malt. Tests for weight per 1,000 kernels, size of kernel, foreign seeds, and presence of mold were featured but little.

The findings given for acrospire growth by the different analysts appear to be in fairly satisfactory agreement with the exception of those given by analysts Nos. 3 and 15 for malt sample No. 1, and for analysts Nos. 2, 3, 4, and 15 for malt sample No. 2.

The variance in bushel weight as reported for malt sample No. 1 was 1.8 lbs., and sample No. 2, 1.1 lbs. These tests are very unsatisfactory for there is no reason why, with proper equipment, test weight per bushel determinations should vary by more than 0.2 lbs.

Terms applicable to the expression of the factor "mellowness of malt," are badly in need of standardization. Eleven different terms were used to describe this condition of the malt. Perhaps half of these terms have a significance, the other half may mean the same thing expressed differently. Efforts should be made to define these terms as well as to identify their significance.

TABLE IV—*Continued*

Collaborator number											Average
8	9	10	11	12	13	14	15	16	17	18	
5.30	6.20	6.24	5.33	6.40	6.40	—	6.78	6.50	6.82	5.90	6.31
—	—	—	—	—	—		—	—	—	—	—
—	—	—	—	—	—		2.34	—	—	—	—
148	—	185	189	192	185		—	167	162 *a/*	—	173
69.28	65.96	67.64	65.41	67.28	66.14		66.45	68.60	—	68.00	67.05
73.36	70.33	72.12	69.09	71.89	70.66		71.28	73.38	—	72.25	71.47
—	—	—	—	—	—		16.77	—	—	—	16.46
—	.11	—	.99	—	—		—	—	—	—	.55
Brilliant	Brilliant	Brilliant	—	Clear	Brilliant		—	Clear	—	Clear	Clear
—	Very pale straw	Pale straw	43 *c/*	—	—		—	—	—	—	—
1.87	1.60	1.90	—	1.60	1.80		—	1.75	—	1.20	1.64
—	Fast	Rapid	—	Rapid	Fast		—	—	—	Rapid	Rapid
—	Aromatic	—	—	—	—		—	—	—	—	—
—	—	—	—	1.02996	—		—	1.03061	—	—	1.03132
—	—	*d/*	—	—	—		—	—	—	—	—
1.0315	—	—	—	—	1.03011		1.03025	—	—	—	1.03079
5	5	5	—	5	5		5-10	5	—	5	5
Meindl-Plato	—	Schultz-Ostermann	—	Schultz-Ostermann	Plato		Plato-Pawlowski	Schultz-Ostermann	—	—	—

No sample sent to collaborator

Weight per 1,000 kernels was reported by only 4 analysts. Variance in this factor can reasonably be accounted for by differences in moisture test results as the 1,000 kernel weights were given on a dry-matter basis.

Malt sizing tests, reported by 3 analysts, were far from satisfactory. From a record of the procedures at hand regarding this determination, it is evident that the apparatus and the size of the sieves must be standardized.

Chemical Determinations

The chemical determinations reported were moisture, extract yield (coarse and fine grist), diastatic activity, and acidity of the wort. Appearance, color, filtration, and conversion time of the wort were also reported on by most of the analysts.

Moisture Determinations

The variation in moisture test results with sample No. 1 in the unground state was from 4.32% to 6.60%, or a spread of 2.28%. With sample No. 1 in the ground condition, the variation was greater, being 3.95% to 6.50%, or a spread of 2.55%. Stated another way, this spread in moisture is the equal of approximately 2% of extract, if the average figure of 69.09% for yield of extract, fine grist, on an "as is" basis, as found by 17 analysts, is taken as correct.

The moisture test results on malt sample No. 2, in the unground state, were slightly more uniform. The spread was from 5.16% to 6.90%, or a variation of 1.74%. However, from a ground standpoint, no improvement was apparent, in fact the data are less uniform as the variation was from 5.30% to 7.70%, or a spread of 2.4%. This spread in moisture content again is equal to approximately 2% of extract.

The fact that no improvement in analytical results obtained by grinding the sample indicates that methods of procedure and not variations in the moisture content of the malt sample was the reason for the non-uniform results. A study of the statements made on moisture testing procedure by the 18 analysts who submitted their methods confirms this. There were 15 different moisture test procedures listed on these 18 reports. These procedures varied with respect to (1) the size of sample taken for the moisture test, (2) type of drying oven employed, (3) duration of heating time and temperature of heating.

The size of samples used was 2, 3, 5, and 10 grams.

The types of drying ovens used were far from uniform. While the electrically heated air-oven was used most, ovens heated by gas and also by steam were reported. In addition, the vacuum oven operated at a 100 mm. vacuum basis—at 98°–100° C., was also used by two analysts.

The time of drying the sample varied from 10 minutes to 5 hours; the majority of the operators drying the test sample for 3 hours. A few analysts reported the returning of the sample to the drying oven for an hour additional heating.

Temperature of drying varied from 98° to 132.5° C.

Most analysts heated their drying ovens over the range from 102°–105° C.; 103°–104° C. apparently being the most frequently used temperature.

Referring to the analytical results, it may generally be said that those analysts who used a vacuum-oven or air-oven operated at 130° C. obtained the highest results.

The intimate correlation which exists between moisture test results and extract yields when expressed on the dry basis (being approximately 1% of the extract yield value for each variation in moisture content of 1%) makes it imperative that a moisture test procedure be developed which is standard in all respects, i.e., size of sample, type of moisture tray, type of oven, heating time, and temperature of heating, as well as desiccator for cooling the samples before weighing, if anything near uniformity in test results is to be expected.

Extract Determinations

Most of the analysts reported extract only on the basis of the fine grist, although a few also reported results on the coarse grist, with of course the accompanying differential between extract yields on the coarse and fine grists.

Examining the extract data obtained by the various analysts with malt sample No. 1, unground, on an as is basis (in which condition the moisture determination does not operate to influence the results), there will be found (if we eliminate the data given by analyst 11, because of his extremely low results) that there was a maximum variation of 2.66% in extract yield. Calculating extract to a moisture-free basis made matters much worse as the maximum variation in the analyses then became 3.73%. The large variance in moisture content results is operative to develop this greater spread.

The results obtained by the analysts from the analysis of sample No. 1, in the ground state, were slightly better than the results obtained with this sample in an unground condition. The maximum variation in results, on the as is basis, was 2.27% as compared with a maximum variation of 2.66% when the analysts ground their own sample.

The extract yields obtained from the analysis of malt sample No. 2 are the reverse, in so far as average accuracy is concerned, of the findings recorded for malt sample No. 1. The maximum variation in results for malt sample No. 2, as is basis, where the sample was ground by the analyst, was 2.54%, while on the ground sample the variation on an as is basis was 3.51%, *i.e.*, a greater accuracy when the analyst ground his own sample. Accuracy was not improved by converting to a dry basis.

Although every effort was made to obtain a uniform and representative sample of the ground malt, there is reason to believe, from the above data, that lack of uniformity in the ground sample may be the cause of the unexpected lack of better results with this material. To state the cause of these differences is indeed a difficult task. The differences in moisture test results in connection with the estimation of extract yield on the dry basis, important as they are, as pointed out above, will not clear up the variations. For example, if the variation in moisture test results as reported by the 18 analysts is eliminated by taking for the moisture content of malt sample No. 1 the average value of 5.38% as found by these 18 analysts, and the extract figure as given by each analyst on an as is basis is calculated to a dry basis using the figure (5.38%) rather than the moisture figures reported by them, there will be little improvement in extract yield agreement. The variation in results using the analysts moisture

figures (data from analyst 11 again being eliminated) is 3.73% compared with 2.81%. For malt sample No. 1, ground, the relationship is 2.81% compared with 3.11%; for malt sample No. 2, 3.38% compared with 2.06%, and for malt sample No. 2, ground, 3.52% compared with 3.75%.

Variability in the Extract Yield Determination

From a study of the methods of analysis used by the different analysts in determining extract yield, it is apparent that most of the causes for variation are centered in methods of procedure.

The procedures for making extract determinations appear to be uniformly the same with respect to the first stage of the method, *i.e.*, heating the mash for 30 minutes at 45° C. using a 1 to 5 mash (50 cc. malt to 250 cc. H_2O). Stirring of the mash, however, during this period is very non-uniform, varying from an occasional stirring to a specified 5-minute-interval stirring to a continuous stirring throughout the entire 30 minutes.

The second stage of the test, *i.e.*, raising the temperature of the mash to 70° C. in 25 minutes, was also completed by the majority of analysts with the same technique, namely, at the rate of 1° per minute.

Continued exposure (third stage) of the mash to 70° C. after this temperature has been reached, is not uniformly the same between analysts. This time interval varied from a specified time interval of one hour down to that time necessary for complete inversion after 70° C. has been reached at the end of the second stage of the test. All sorts of continued heating times, from those just mentioned to a 15, 30, and 55 minute interval, were reported. Stirring during this third stage of the test is likewise not standardized.

The fourth stage, cooling of the mash to make up to weight (450 g.), was likewise very non-uniformly carried out. The cooling temperatures reported were 15°, 17.5°, 20°, 25° C., and room temperature. As variations in temperature, in a small way at least, control the volume necessary to build up to a 450 g. mash weight, a standard temperature should be selected for this phase of the extract yield determination.

From the fourth stage on in the extract yield determination non-uniform procedures appear to prevail. Specific gravity determinations were made at 15, 17.5, and 20° C.; pycnometers varied in style and capacity; while 10 different conversion charts were used to convert specific gravity of wort into extract yield.

There are good indications that the conversion table situation is the greatest source of error in reporting extract yield data, and that

some of the errors reported above could be reduced if a standard temperature for making specific gravity determinations could be agreed upon and a standard conversion table used for determining extract yield. This will be apparent if the specific gravity values given by analysts 5, 6, 7, 8, 13, 14, and 15 are taken and a single standard conversion chart used to compare extract yield data. The variance in this example is approximately 1%.

To the chairman, at least, lack of uniformity in the stirring procedure during the several stages of the extract determination is probably the next important source of error, with differences in size and capacity of pycnometer assuming third place in importance.

Diastatic Activity

Variations in returns on diastatic activity determination were outstandingly great. However, the reasons for these variations seem to be more apparent than with the determination for extract. Further, if the results obtained by those analysts who did not use a buffer solution are segregated from those who did use a buffer solution, the variations between different analysts are not so significant. In other words, where great differences in results between analysts occurred it was with those cases where no buffer solutions were used. The next important source of error seems to be localized in the different procedures for making the test. One group of analysts, for instance, determines diastatic activity by reducing varying quantities of Fehling's solution with a constant and uniform quantity of converted starch solution; while a second important group maintains the Fehling's solution as a constant and varies the amount of converted starch solution. A third group, notably analysts 2 and 17, while making the malt infusion in the same manner as the other analysts, use copper reduction methods quite different from the other analysts.

Reasonably close agreements for time of conversion, appearance, and filtration of wort were reported by all analysts making these determinations.

Three different methods for expressing color of wort were reported, *i.e.*, terms Lovibond units, Stamner units, and as cubic centimeters of N/10 iodine solution, the majority however reporting in terms of Lovibond units. With the Lovibond procedure, reasonably close agreements in the reporting of results were experienced.

These results were again presented to interested malt and brewing chemists at a second meeting in Chicago in December.

At this period in the history of the A. A. C. C. malt committee there was formed by the three large interests who are mainly concerned

TABLE V

RESULTS OF COLLABORATIVE ANALYSIS ON MALT SAMPLE NO. 1 (DETERMINED ACCORDING TO THE METHODS OF THE M. A. S. C. ADOPTED JAN. 14, 1935, CHICAGO, ILL.)

A. Physical Analysis

Collaborators' Numbers and Letters

Determinations		1	2	4	6	7	8	9	10	11	12	13	14	16	17	G	A	B	C	D	E	F	Average
Bushelweight, *lbs.*		—	38½	38½	—	37	—	37¾	37½	38	—	37½	36¾	37¾	38½	37½	36¾	37	—	37¼	—	38	37.6
1000 Kernel weight, *gms.*	As is	42.2	42.5	41.7	39.7	42.8	—	42.8	41.4	42.1	42.0	42.4	42.2	42.4	42.5	—	43.0	42.9	—	42.3	—	—	42.2
	Dry basis	40.0	40.2	39.4	37.6	40.5	—	40.5	39.3	40.2	39.8	40.2	40.0	40.2	40.5	—	40.7	40.6	—	40.3	—	—	40.0
Growth, length of acrospire	0–1/4	6	1	3	4	4	6	3	2	2	3	2	2	2	2	3	3	13	—	2	—	2	3.4
	1/4–1/2	4	1	9	4	3	0	5	3	5	5	2	3	4	6	5	2	3	—	2	—	2	3.6
	1/2–3/4	12	3	45	8	7	0	28	5	12	11	5	5	19	12	6	20	6	—	16	—	7	11.9
	3/4–1	77	95	40	78	86	93	64	89	78	79	90	89	69	80	85	70	78	—	80	—	89	79.4
	Over	1	0	3	6	0	1	0	1	3	2	1	1	6	0	1	5	0	—	0	—	0	1.6
Mealiness, %	Mealy	99	100	79	94	89	X	100	97	92	98	95	94	98	96	95	91	90	—	98	—	96	94.5
	Half-glassy	1	0	16	2	10	—	0	3	6	2	3	6	2	4	5	9	7	—	2	—	4	4.6
	Glassy	0	0	5	4	1	—	0	0	2	0	2	0	0	0	0	1	3	—	0	—	0	1.0
Foreign seeds and broken kernels, %		0.6	—	0.2	0.0 0.1	.46	— X —	— X —	— — —	0.3	X	0.2	0.04 0.2	—	X	.30	—	—	—	.15	—	—	.3
Assortment, %	on 7/64"	88.3	95.2	—	—	—	—	95.8	94.8	96.0	—	95.3	—	—	—	—	—	—	—	—	—	97.8	94.7
	on 6½/64"	10.0	4.6	—	—	—	—	—	4.6	3.5		4.6	—	—	—	—	—	—	—	99.7*	—	2.0	4.9
	on 5½/64"	1.0	0.2	—	—	—	—	—	0.3	0.1	—	0.1	—	—	—	—	—	—	—	.1*	—	.2	.3
	on 5/64"	0.0	0.0	—	—	—	—	—	0.2	0.1	—	0.0	—	—	—	—	—	—	—	.1*	—		Tr
	through 5/64"	0.7	0.0	—	—	—	—	—	0.1	—	—	0.0	—	—	—	—	—	—	—	.1*	—		Tr
Mold		X	None	None	None	None	None	None	—	None	None	None	None	None	None		None	—	—	Tr*	—	None	None (93%)

TABLE V—*Continued*

B. Chemical Analysis

Moisture, %		5.3	5.4	5.6	5.3	5.4	5.0	5.4	5.1	4.5	5.2	5.1	5.1	5.2	4.6	4.8	5.4	5.4	4.6	4.8	5.0	5.0	5.1
Extract in the finely ground malt (Plato), %	As is	73.0	72.3	72.1	72.1	72.8	73.2	72.8	72.7	72.5	72.2	72.7	73.3	72.5	72.7	71.9	71.9	72.6	71.4	72.5	72.5	73.2	72.5
	Dry basis	77.1	76.5	76.4	76.0	77.0	77.1	77.0	76.6	75.9	76.2	76.7	77.2	76.5	76.2	75.5	76.0	76.7	74.8	76.2	76.3	77.1	76.4
Conversion in minutes		7–10	Less than 5	10–15	10–15	5–7	X	7–10	5–7	7–10	15–20	10–15	Less than 5	Less than 5	Less than 5	7–10	7–10	7–10	8	Less than 5	10	10	—
Color of lab. wort Lovibond tint. ¼" cell Ser. 52		1.6	1.5	—	—	1.6	1.8	1.6	—	1.5	1.5	1.7	1.6	1.5	1.8	1.2	—	—	1.5	1.4	—	1.5	1.56
Odor of mash		s.a.	s.a.	a	a	s.a.	—	X	—	s.a.	a	a	a	X	a	a	a	s.a.	s.a.	s.a.	—	a	a—44% s.a.—56%
Degree of clarity		h	s.h.	h	h	s.h.	h	h	h	s.h.	h	s.h.	h	h	s.h.	c	s.h.	h	s.h.	s.h.	—	s.h.	h—50% s.h.—45%
Speed of filtration		sl	n	n	n	n	—	n	n	sl	n	n	n	n	n	n	n	n	n	n	—	n	n (89%)
Extract in the coarsely ground malt (Plato), %	As is	72.8	70.8	71.9	—	72.0	—	—	72.1	72.1	—	72.3	72.1	—	—	—	—	—	—	—	—	—	72.0
	Dry basis	76.9	74.6	76.1	—	76.1	—	—	76.0	75.5	—	76.3	76.0	—	—	—	—	—	—	—	—	—	75.9
Diastatic power, degree Lintner		64	68	—	—	65	65	—	—	62	—	62	67	—	77	61	90	72	69	69	73	—	69.0

* Not included in average.

Key to abbreviations:

X = Not determined according to M. A. S. C. standards.

Degree of clarity—
h = hazy
s.h. = slightly hazy
c = clear.

Odor of mash—
a = aromatic
s.a = slightly aromatic.

Speed of filtration—
n = normal
sl = slow.

TABLE VI

RESULTS OF COLLABORATIVE ANALYSIS ON MALT SAMPLE NO. 2 (DETERMINED ACCORDING TO THE METHODS OF THE M. A. S. C. ADOPTED JAN. 14, 1935, CHICAGO, ILL.)

A. Physical Analysis

Collaborators' Numbers and Letters

Determinations		1	2	4	6	7	8	9	10	11	12	13	14	16	17	G	A	B	C	D	E	F	Average
Bushelweight, *lbs.*		—	38¼	38	—	37	—	37½	37¼	37½	—	.37	35½	37.0	38.0	37	37	36½	—	36¾	—	37¾	37.2
1000 Kernel weight, *gms.*	As is	32.7	33.1	33.0	31.4	32.4	—	33.0	32.6	33.2	34.0	33.4	33.7	32.3	33.0	—	32.9	33.2	—	32.9	—	—	32.9
	Dry basis	31	31.4	31.3	29.8	31.0	—	31.3	31.0	31.6	32.2	31.6	32.1	30.6	31.5	—	31.0	31.5	—	31.4	—	—	31.3
Growth, length of acrospire	0–1/4	8	3	5	2	2	2	1	3	0	3	3	1	1	2	5	2	6	—	2	—	0	2.7
	1/4–1/2	0	1	6	2	3	0	2	3	3	2	2	2	3	6	3	3	4	—	0	—	0	2.4
	1/2–3/4	3	1	23	4	10	1	8	5	1	5	5	2	18	8	3	10	7	—	8	—	5	6.7
	3/4–1	82	92	51	88	77	88	88	87	84	81	89	95	75	82	84	77	80	—	86	—	90	82.9
	Over 1	7	3	15	4	8	9	1	2	12	9	1	0	3	2	5	8	3	—	4	—	5	5.3
Mealiness, %	Mealy	98	98	72	90	71	X	96	89	87	93	92	92	90	92	95	71	82	—	96	—	90	88.6
	Half-glassy	2	1	23	8	27	X	4	9	12	5	6	8	9	8	4	27	15	—	4	—	8	10.0
	Glassy	0	1	5	2	4	X	0	2	1	2	2	0	1	0	1	4	3	—	0	—	2	1.7
Foreign seeds and broken kernels, %		0.4	—	0.1	0.0 0.4	—	X X	X	—	0.5	X	0.3	— 0.2	— —	X	.4	—	—	—	.19	—	—	.3
Assortment, % on 7/64″		62.0	80.5	—	—	—	—	83.2	74.6	81.2	—	77.5	—	—	—	—	—	—	—	95.1*	—	89.6	78.4
on 6½/64″		26.0	16.2	—	—	—	—	—	19.9	15.2	—	19.1	—	—	—	—	—	—	—	3.9*	—	8.6	17.5
on 5½/64″		10.7	2.6	—	—	—	—	—	3.4	2.9	—	2.8	—	—	—	—	—	—	—	.6*	—	1.2	8.2
on 5/64″		1.0	0.5	—	—	—	—	—	1.4	0.3	—	0.4	—	—	—	—	—	—	—	.4*	—	.5	.7
through 5/64″		0.7	0.2	—	—	—	—	—	0.7	0.1	—	0.2	—	—	—	—	—	—	—		—	.1	.3
Mold		X	None	None	None	None	None	None	—	None	None	None	None	None	None	None	None	—	—	Tr*	—	None	None (94%)

TABLE VI—*Continued*

B. Chemical Analysis

Moisture, %		5.2	5.2	5.3	5.0	5.1	5.0	5.1	5.0	4.4	5.1	5.2	5.0	5.2	4.5	4.9	5.6	5.2	4.5	4.6	5.5	4.9	5.0
Extract in the finely ground malt (Plato), %	As is	70.7	69.2	69.6	70.2	70.3	70.2	70.5	69.9	70.3	69.8	70.0	70.0	69.7	70.0	69.8	69.6	70.0	68.6	70.4	69.9	69.3	69.9
	Dry basis	74.6	73.0	73.4	73.9	74.1	73.9	74.3	73.6	73.5	73.5	73.9	73.7	73.6	73.3	73.4	73.7	73.4	72.0	73.8	74.0	72.9	73.6
Conversion in minutes		7–10	Less than 5	7–10	7–10	Less than 5	Less than 5	5–7	Less than 5	5–7	5–7	7–10	Less than 5	Less than 5	Less than 5	Less than 5	Less than 5	5–7	5	Less than 5	6	5	—
Color of lab. wort Lovibond tint. ½" cell, Ser. 52		1.5	1.5	—	—	1.6	1.6	1.6	1.6	1.3	1.3	1.5	1.5	1.5	1.7	1.5	—	—	1.5	1.4	—	1.5	1.5
Odor of mash		s.a.	s.a.	s.a.	s.a.	s.a.	—	X	—	s.a.	a	s.a.	a	X	s.a.	a	s.a.	s.a.	s.a.	s.a.	—	a	a—25% s.a.—75%
Degree of clarity		h	s.h.	s.h.	s.h.	c	c	s.h.	s.h.	c	s.h.	s.h.	s.h.	s.h.	s.h.	c	c	s.h.	c	c	—	c	c—35% s.h.—55%
Speed of filtration		n	n	n	n	n	—	n	n	n	n	n	n	n	n	n	n	n	n	n	—	n	n (100%)
Extract in the coarsely ground malt (Plato), %	As is	69.5	66.7	68.7	—	—	—	—	68.8	69.2	—	69.4	68.4	—	—	—	—	—	—	—	—	—	68.7
	Dry basis	73.3	70.4	72.6	—	—	—	—	72.4	72.4	—	73.3	72.0	—	—	—	—	—	—	—	—	—	72.3
Diastatic power, degree Lintner		150	149	—	—	150	135	—	—	140	—	141	147	—	178	139	188	151	156	137	172	—	152

* Not included in average.

Key to abbreviations:
X = Not determined according to M. A. S. C. standards.

Degree of clarity—
h = hazy
s.h. = slightly hazy
c = clear.

Odor of mash—
a = aromatic
s.a. = slightly aromatic.

Speed of filtration—
n = normal.

TABLE VII

RESULTS OF COLLABORATIVE ANALYSIS ON MALT SAMPLE NO. 3 (DETERMINED ACCORDING TO THE METHODS OF THE M. A. S. C. ADOPTED JAN. 14, 1935, CHICAGO, ILL.)

A. Physical Analysis

Collaborators' Numbers and Letters

Determinations		1	2	4	6	7	8	9	10	11	12	13	14	16	17	G	A	B	C	D	E	F	Average
Bushelweight, *lbs.*		—	37½	38.0	—	37	—	38.0	37½	37½	—	37	36¼	36½	37¾	37	36½	37	—	36¾	—	37	37.1
1000 Kernel weight, *gms.*	As is	—	23.9	23.8	22.3	23.4	—	23.8	23.6	23.8	22.8	23.6	23.5	23.8	23.9	—	23.9	23.7	—	23.4	—	—	23.5
	Dry basis	—	22.8	22.7	21.3	22.3	—	22.7	22.5	23.0	21.8	22.5	22.4	22.6	22.9	—	22.6	22.6	—	22.5	—	—	22.5
Growth, length of acrospire	0–1/4	3	1	3	2	3	3	6	3	2	3	3	4	1	2	3	1	5	—	4	—	4	3.0
	1/4–1/2	1	1	5	2	1	0	3	2	1	4	3	5	3	10	4	3	0	—	0	—	0	2.5
	1/2–3/4	12	1	28	4	2	0	13	5	3	9	3	4	23	18	3	10	10	—	10	—	8	8.7
	3/4–1	83	96	54	86	92	92	77	87	83	80	90	87	69	70	87	83	83	—	80	—	81	82.1
	Over	1	1	10	6	2	5	1	3	11	4	1	0	4	0	3	3	2	—	6	—	7	3.7
Mealiness, %	Mealy	—	98.0	59	78	81	X	93	90	74	92	81	90	83	92	92	72	72	—	94	—	88	84.0
	Half-glassy	—	2.0	21	8	17	X	6	9	20	7	16	8	13	8	6	24	21	—	4	—	8	11.6
	Glassy	—	0.0	20	14	2	X	1	1	6	1	3	2	4	0	2	4	7	—	2	—	4	4.4
Foreign seeds and broken kernels, %		1.0	—	0.2	0.1 0.5	.74	X	X	—	0.8	X	0.4	0.2 0.2	—	X	.4	—	—	—	.66	—	—	.55
Assortment, %	on 7/64"	4.5	19.2	—	—	—	—	19.2	10.9	18.8	—	16.4	—	—	—	—	—	—	—	—	—	33.2	17.4
	on 6½/64"	21.5	49.2	—	—	—	—	—	51.7	44.7	—	52.0	—	—	—	—	—	—	—	57.7	—	49.6	46.6
	on 5½/64"	58.0	28.3	—	—	—	—	—	28.8	31.5	—	27.9	—	—	—	—	—	—	—	34.1	—	15.2	31.9
	on 5/64"	12.0	2.7	—	—	—	—	—	6.8	3.3	—	2.8	—	—	—	—	—	—	—	5.8	—	1.6	5.0
	through 5/64"	5.0	0.6	—	—	—	—	—	1.8	0.9	—	0.9	—	—	—	—	—	—	—	2.4	—	.4	1.7
Mold		X	None	None	None	None	None	None	—	None	None	None	None	None	None	None	Tr	—	—	Tr	—	None	None (88%)

TABLE VII—*Continued*

B. Chemical Analysis

Moisture, %	4.7	4.4	4.7	4.6	4.7	4.4	4.6	4.6	3.5	4.6	4.6	4.5	4.7	4.3	4.5	5.2	4.8	3.7	3.9	4.7	4.3	4.5
Extract in the finely ground malt (Plato), % As is	69.5	67.8	68.5	68.7	69.3	68.3	69.3	68.8	68.8	68.7	68.7	68.6	68.3	69.0	68.5	68.8	68.7	66.2	69.0	69.2	68.5	68.7
Dry basis	72.9	70.9	71.8	72.0	72.7	71.4	72.6	72.1	71.3	72.0	72.0	71.8	71.7	72.1	71.7	72.4	72.2	68.7	71.8	72.5	71.6	71.8
Conversion in minutes	7–10	Less than 5	5–7	7–10	Less than 5	Less than 5	Less than 5	Less than 5	—	Less than 5	Less than 5	Less than 5	Less than 5	Less than 5	Less than 5	Less than 5	Less than 5	5	Less than 5	6	Less than 5	—
Color of lab. wort Lovibond tint. ¼" cell, Ser. 52	1.8	1.9	—	—	1.7	1.8	1.8	1.8	1.8	1.3	1.7	1.5	1.5	1.7	1.6	—	—	2.0	1.6	—	1.7	1.7
Odor of mash	s.a.	s.a.	s.a.	s.a.	s.a.	—	X	—	s.a.	a	s.a.	a	X	s.a.	a	s.a.	s.a.	a	s.a.	—	a	a—29% s.a.—71%
Degree of clarity	h	c	c	c	c	c	c	c	c	c	c	c	s.h.	c	c	c	c	c	c	—	c	c—90%
Speed of filtration	n	n	n	n	n	—	n	n	n	n	n	n	n	n	n	n	n	n	n	—	n	n—100%
Extract in the coarsely ground malt (Plato), % As is	68.1	65.6	67.7	68.3	—	—	—	67.6	67.8	—	68.3	67.4	—	—	—	—	—	—	—	—		67.6
Dry basis	71.5	68.5	71.1	71.5	—	—	—	70.9	70.2	—	71.6	70.5	—	—	—	—	—	—	—	—		70.7
Diastatic power, degree Lintner	150	154	—	—	150	140	—	—	146	—	145	162	—	197	151	202	154	169	153	151	—	158

Key to abbreviations:
X = Not determined according to M. A. S. C. standards.

Degree of clarity—
h = hazy
s.h. = slightly hazy
c = clear.

Odor of mash—
a = aromatic
s.a. = slightly aromatic.

Speed of filtration—
n = normal
sl = slow.

with the manufacture and usages of malt, a committee known as the Malt Analysis Standardization Committee (Chicago). This committee was composed of representatives of the large malt houses, large breweries, and professional laboratories which offer advice to the malting and brewing industry. Due to the chaotic condition in the industry due to the lack of standard methods for the analysis of malt, this committee was charged with the responsibility of working up, in a rapid way, a set of methods which could be adopted by the malting and brewing industry as a set of official methods for their purposes. This committee released their methods in booklet form in January 1935, and subsequently carried out a series of collaborative tests by the new methods amongst their personnel.

The A. A. C. C. malt committee was invited to collaborate in this study. The results of the tests which were experienced using the Malt Analysis Standardization Committee's (Chicago) procedures on three samples of malt are given in Tables V, VI, and VII. The numerical numbers represent collaborators who were members of the Malt Analysis Standardization Committee (Chicago) while the alphabetical designations refer to members of the A. A. C. C.

It is apparent from an examination of these data that there is considerably less variance in analytical results when using the procedures sponsored by the Malt Analysis Standardization Committee (Chicago), than was the case when all collaborators used methods characteristic of their own laboratories, such as is illustrated in Tables I, II, III, and IV.

It is not the practice of the A. A. C. C. committees to be stampeded into accepting as official methods of analysis which may on the surface appear meritorious, without first undertaking an extensive study of their efficiency and reliability. Illustrative of the manner in which the malt committee plans to study the various methods for evaluating malt is the extensive work done by the chairman in the instance of the moisture test method for malt, the findings on which will be found in another article in this issue. This committee plans to subsequently examine the methods sponsored by the Malt Analysis Standardization Committee (Chicago) for such determinations as extract yield, diastatic activity, etc., in the same manner as for the moisture test study.

There is this to be said of the extract yield procedure of the Malt Analysis Standardization Committee (Chicago) which is well worth while, namely, that it has been evolved on the basis of the procedure described in the Salzburg Agreement, which has been in use in Europe for a considerable period of time with satisfaction. With the standard equipment being adopted for use in carrying out this method, entirely satisfactory results should be obtained by its use.

The diastatic activity method of the Malt Analysis Standardization Committee (Chicago) is, in effect, that which has been used successfully by the Institute of Brewing (British) for some time, and it is believed therefore that it has all the requisites of an efficient standard method.

Recommendations

As a result of the first year's work, the Committee recommends:

1. That the tentative methods of analysis as adopted by the Malt Analysis Standardization Committee (Chicago) be adopted by this Association as tentative methods for study.

2. That the moisture determination method as described by the Malt Analysis Standardization Committee (Chicago) be adopted as a tentative official method by this Association and that further work be done to confirm it as an official method.

BARLEY AND MALT STUDIES: I. DEVELOPING NEW VARIETIES OF BARLEY FOR MALTING AND THEIR PROPERTIES [1]

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(Read before the American Association of Cereal Chemists at Denver, Colorado, June 7th, 1935.)

Introduction

The discussion given in this paper will consist largely of methods of procedure in a coordinated program for barley improvement. This program must of necessity take into consideration the interests of the producer of the crop as well as the consumer. The data upon which this discussion of quality in barley is given, are preliminary, based largely upon one year's results. They are given therefore only to illustrate certain needs and must not be taken as final in relation to barley varietal quality or in relation to methods of procedure in determining quality in barley.

Perhaps it is wise to define our point of view in relation to producing, handling, marketing, and using the barley crop before proceeding to the discussion of the subject assigned. Certainly much can be done immediately in improving the quality of the barley available by intelligently planning and executing an educational program among farmers, elevator men, and others in helping recognize barley quality and developing methods to insure proper financial recognition and delivery in a natural state to the ultimate consumer. This phase of the problem has been discussed before farm groups, the elevator and grain associations, and the consumer's organizations, and is mentioned here only to emphasize that any program in developing improved varieties of barley is immaturely launched until there is some unanimity of opinion and action regarding these economic phases of the problem.

All are agreed that we need improved and more uniform quality in

[1] Based on cooperative investigations between the Division of Cereal Crops and Diseases, Bureau of Plant Industry, United States Department of Agriculture and the Wisconsin Agricultural Experiment Station. The cooperative investigations include the agricultural experiment stations of Colorado, Illinois, Iowa, Michigan, Minnesota, Montana, Nebraska, North Dakota, Ohio, and South Dakota where the uniform barley varietal series are grown each year. The United States Maltsters Association has cooperated through an industrial fellowship grant to the University of Wisconsin.

barley although at present no two groups appear to agree on what constitutes quality in the broader sense of the term. The producer is interested in high yields, economy in handling the crop and net returns from the grain and straw. The consumer is interested principally in quality in relation to the manufacturing processes and the products, although indirectly he is interested or should be interested, in the methods and economy of production. The coordination of the programs of these different interests is perhaps as big a problem as the production of a desirable malting barley.

The malting barley for the central and eastern market is produced largely in the more humid North Central United States. In general, the farmers throughout this area are growing barley for feed and as a cash crop. A high yielding variety of good malting quality will enable the grower to produce, at a minimum cost, a barley which may be sold as a cash crop when quality and prices are satisfactory or which may be fed on the farm. Growing two varieties of barley, one for feed and one for the malting market, is not practical on the average farm throughout this region. The reasons are obvious. Mixtures of the two varieties are inevitable in the ordinary procedure of threshing, binning and marketing of the grain. The flexibility of the seasonal supply of high quality barley as well as the farmer's opportunity of taking advantage of the differential between the prices of feed and malting barley is limited. Therefore, at the outset, we will consider the development of new barley varieties from the standpoint of the dual purpose, feed and malt. This means the variety must be adapted to the area in which it is grown, give high yields, be resistant to the principal diseases of the crop, be satisfactory from the standpoint of ease in handling as well as utilization of the straw and at the time contain the desirable attributes making up malting quality.

A brief survey of the average annual use of the barley crop will help illustrate the point of view briefly defined and give some indication of the quality needed in industrial barley. The chart given in Figure 1, showing the estimated average barley production in the United States, barley fed to livestock, barley exported and imported and barley pearled and used for domestic malting for 1914, 1929 and 1931 indicates that about 38% of the crop was used for malting in 1914, somewhat less than 9% in 1929, and about 12% in 1931. The barley used for malting in 1934–35 is estimated by the trade associations at about 65 million bushels which would amount to about 30% of the barley produced based on an average crop. This amount is certainly sufficient to warrant concerted effort on the part of all interested in barley toward the production of satisfactory malting quality in the areas adapted to the production of malting barley. Inasmuch as approx-

imately 86% of the malt produced is used for brewing purposes in contrast to 9% for the production of diastatic malt and 5% for malt used in other processes, chief emphasis should be placed on quality

Fig. 1. Graph showing barley produced and its use in the United States. Data taken from the United States Department of Agriculture Year Books, U. S. Census Reports and Industrial Reports of Trade Associations. The barley used for malting in 1934–35 is estimated by the trade associations at about 65 million bushels which would amount to about 30% of the barley produced based on an average crop.

in relation to malt for brewing purposes where the quality requirements are perhaps both more exacting and less specifically defined.

Expansion of Barley Production in the United States

Early production of barley was concentrated largely around the principal malting centers. See Figure 2. The production was chiefly

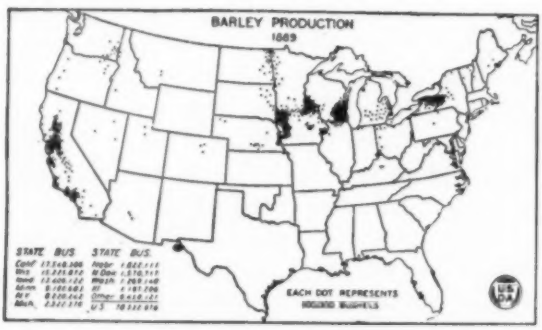

Fig. 2. Barley production in the United States in 1889 showing centralization principally around the malting centers and the export center in California. Taken from U.S.D.A. Dept. Bul. 1334.

for malting purposes with relatively little used for feed. Later with the recognition of the feeding value of the grain and the development of higher yielding varieties, the acreage in barley expanded including

territory where the conditions were less favorable for producing barley of good malting quality. See Figure 3.

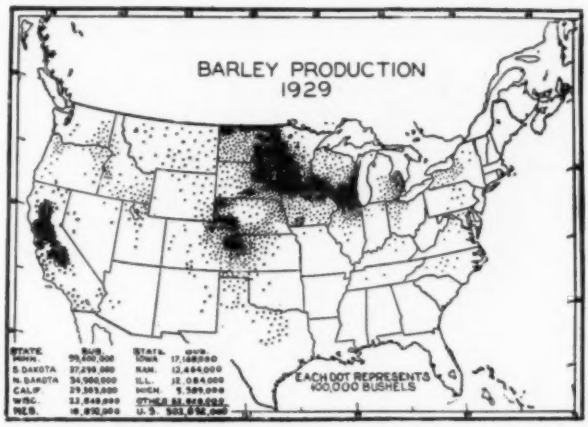

Fig. 3. Barley production in the United States in 1929 showing the expansion of the acreage with the increased use of barley as a feed for livestock and the introduction of new high yielding varieties. Data taken from the U.S.D.A. Year Books.

Survey of Barley Varieties Available

The six-rowed type of barleys have predominated in the United States throughout the period of barley culture. Broadly speaking, the six-rowed varieties may be divided into two general groups; the Manchuria types of North European and Asiatic origin and the Coast types of North African origin. The former are grown throughout the Northeast and Central United States, the latter predominantly in the California district although they extend over into the South Central Rocky Mountain and Great Plains areas. The Manchuria-Oderbrucker types almost exclusively are used for malting purposes in the United States and Canada although considerable quantities of the California barleys have been malted during the past two years. The general regions of adaptation of the various varietal types are shown in Figure 4.

The Manchuria type was first introduced into the United States about 1861. The Manchuria variety or selections from it are grown extensively in Minnesota, the Dakotas and Canada. O.A.C. 21 a selection from Manchuria with gray-blue kernels is grown predominantly for malting in Canada. Oderbrucker, a white kerneled selection from Manchuria, is better adapted to the central and southern portion of the humid spring barley section. Odessa from the same general group as the Manchuria is adapted to the central and south central Great Plains area.

More recently varieties of hybrid origin have been grown extensively in this area. Notable varieties of hybrid origin are Velvet and Glabron originating from crosses between Manchuria and the black,

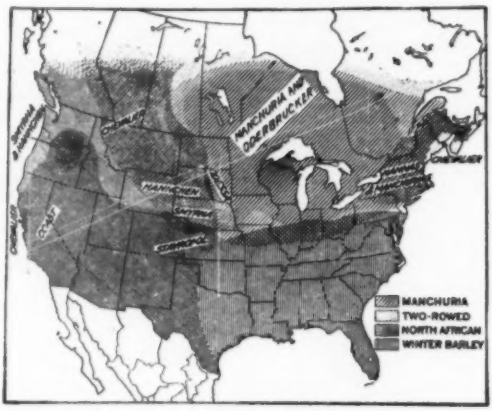

Fig. 4. Map of the United States and Canada showing the general areas to which groups of barleys are adapted with no relation to acreage or production. Taken from U.S.D.A. Dept. Bul. 1334.

smooth awned variety Lion, and Wisconsin Barbless Ped. 38 from a cross between Oderbrucker and a selection from the variety *nigrum leiorrhynchum* similar to Lion. While these hybrid varieties are similar in many characteristics to the Manchuria and Oderbrucker varieties, they differ in other respects. These hybrid varieties met with considerable favor on the part of the producer because of their smooth awns and in some cases higher yields.

The Trebi variety, somewhat similar to the Coast type, originating from a selection made from an introduction of barley from Asiatic Turkey, is well adapted to the drier northern areas of the Great Plains and west of the Rocky Mountains. This variety has given very high yields through these sections and as a result has replaced considerable acreage formerly producing the Manchuria type.

Malting Quality and the Need for Research

Barley was produced largely as a feed grain during the period of national prohibition. The gradual increase in barley production in the United States from 1918 to 1932 was largely for feed purposes. The old standard malting varieties were replaced in part by new varieties of relatively unknown malting quality. The method of handling the crop had changed resulting in more damaged barley than in the years of specialized malting barley production. The grain was marketed with little attention paid to maintaining the identity of the variety or the quality of the barley. Inter-area and inter-market

exchange of barley had increased with the expansion of barley production into the less humid sections. All of these factors combined with unfavorable weather and soil conditions were responsible for the relatively limited amount of good malting barley shipped to market during the past two or three years.

Research on malting quality in barley and testing new varieties for malting quality was needed in reorganization of the barley production program especially in the North Central section of the United States. The experiment station and federal research and extension men interested in barley production formulated a research coordinating organization under the Barley Improvement Council through which eleven states are cooperating. The United States Maltsters Association entered into the cooperative research organization two years ago and has contributed generously both in facilities and funds. In addition, certain industrial laboratories have cooperated in the work. We feel that other organizations interested in research on barley quality will join in making this a more complete and permanent research program.

Malting Equipment and Procedure Used in the Preliminary Study of Malting Quality

Experimental malting equipment is available at several locations for the survey of the malting quality of the barley varieties and testing new selections. The equipment at Madison, Wisconsin, which is designed for handling 15 to 20 pound samples, is briefly described. The malting chamber is essentially similar in general principles to a compartment malt house. The twenty-four individual compartments are placed in hoppers in the malting chamber. The air is passed through the tempering chamber above the malting chamber after which it is drawn down through the barley. Air temperature and moisture content of the air are controlled by a thermostated cooling unit and water sprays. The temperature is held at about 15–16° C. (60.8° F.). The moisture content of the air is maintained slightly below the saturation point. The tempered air is drawn through each compartment at a rate which insures uniformity in temperature throughout the barley mass during the germination period. The malting chamber is shown in Figure 5 with some of the compartments in place and one on the rod ready for turning the malt.

The most suitable procedure for the comparative malting of the barley samples is briefly outlined. The barley samples are cleaned uniformly to remove the small kernels and foreign materials. A preliminary or pilot steep, run on small samples of each lot of barley prior

to malting, is used to determine the approximate time necessary in the steep tank to reach the desired moisture content. This is done so that a steeping schedule can be worked out to remove all of the compart-

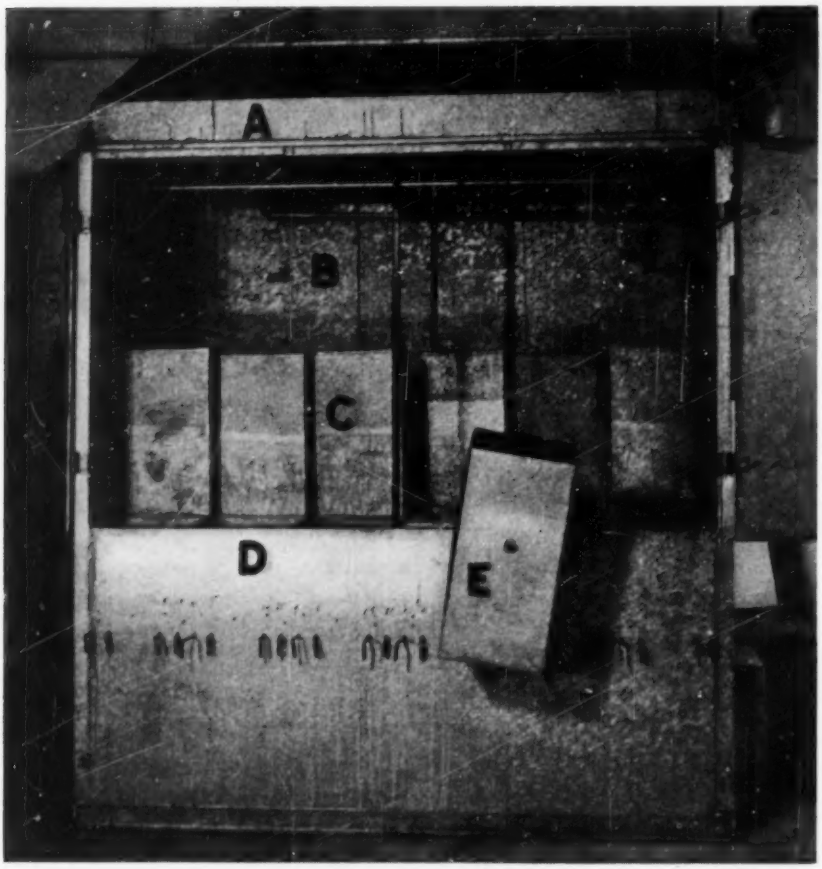

Fig. 5. Front view of the experimental malting chamber with the doors open and the cans in place in the hoppers. The air tempering and humidifying chamber (A) is situated above the malting chamber (B). The individual compartments are shown at C. The hoppers with dampers in the outlets are shown at (D). One compartment (E) is on the rod ready for turning the growing barley.

ments from the steep tank at approximately the same time. A fifteen pound sample of each barley to be malted is placed in a tared malting compartment. The compartments are then placed in the steep tank at the proper time intervals to have all barley lots reach about 42–45% moisture content at the end of the steeping period. The water, maintained at 15° C. (59° F.), is changed during steeping every 8–12 hours. Each compartment is drained and weighed at the end of the steeping period and all are placed in the malting chamber at the same

time. The samples are grown in the malt chamber with constant circulation of the tempered air until the coleoptile or acrospire is approximately the full length of the kernel. This generally requires six days. The compartments are removed and the barley turned every eight hours during the period in the chamber. The compartments are weighed each morning and water added to maintain the desired moisture content after which the barley is turned to secure thorough distribution of the water. The moisture content of the barley at the end of the growing period is about 46–49%. The drying is started at a relatively low temperature; 30° C. (86° F.), after about six hours the temperature is raised to 50° C. (122° F.) and drying is finished at 70°–80° C. (158–176° F.). The rate of drying is appreciably more rapid than in commercial practice due to the high air velocity. The dried samples of malt are weighed, the roots removed and cleaned, reweighed and sampled before storing.

A number of experimental maltings have been made with several varieties of barley using different moisture contents of the barley at the end of the steep period and during the period of growth in the chamber. Space will not permit a detailed discussion of the results. In general, however, there may be a range of 4 to 6% moisture content in steeping and 3–4% moisture content in growing without appreciable effect, based upon the determinations made thus far upon the malt of different experiments. The barley malted at the higher moisture contents gives a lower recovery of malt, slightly lower extract, no

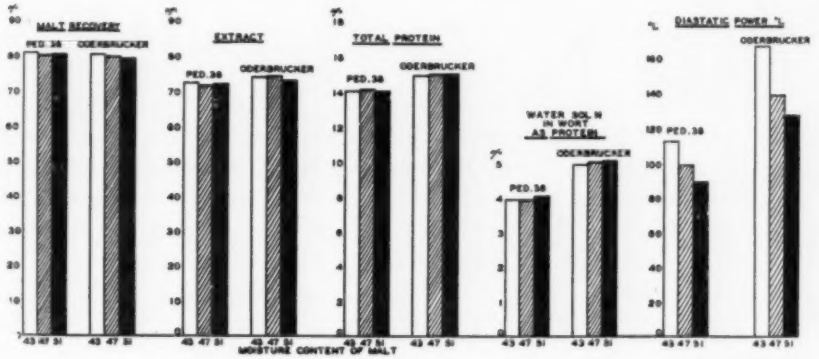

Fig. 6. Graph showing the influence of moisture on malting Ped. 38 and Oderbrucker barleys grown in the season of 1934. Average moisture content at the end of the growing period in the malt chamber is indicated below each bar: 43, steeped to 45% moisture, malted at 43% moisture. 47, Steeped to 45% moisture, malted at 47% moisture. 51, Steeped to 50% moisture, malted at 51% moisture.

appreciable change in total protein, and an increase in water soluble nitrogen in the wort. The diastatic power of the malts was highest at the lower moisture used in steeping and growing. See Figure 6.

Malts produced in the experimental unit have been compared with commercial malts produced from the same barley. Comparisons have been made with three different commercial plants, two with compartment houses and one with drum units. The summary of these comparisons are given in Figure 7. The results have been remarkably

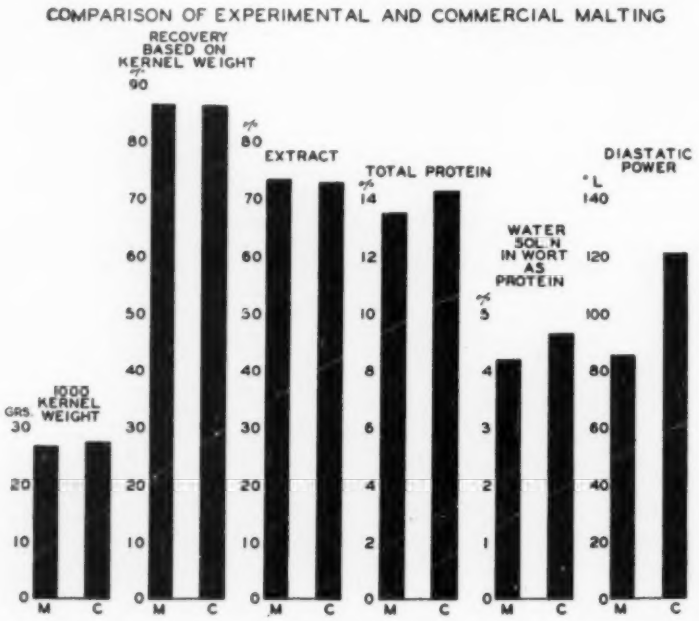

Fig. 7. Graphs showing the comparison of experimental and commercial malting. The bars on the left with (M) below, indicate the determinations on the malts in the experimental units at Madison: those on the right, with (C) below, the commercial malts. The graph is prepared from the average of malts produced from twelve different barleys.

similar insofar as malt recovery and yield of extract are concerned. The total protein and water soluble nitrogen in the wort has been slightly higher in the malts from the commercial units. The diastatic power of the malts likewise has been higher in the commercial malts especially in those with high diastatic power. With the exception of the drying procedure which needs further modification the experimental malting seemingly gives results comparable with commercial production.

Further investigations are anticipated using smaller samples of barley in compartments one-fourth the size of the present units. The malting of smaller samples, if successful, will enable testing selections before large quantities of barley are produced.

Comparative Varietal Studies

The results obtained in this study are too limited to be used in the final evaluation of the malting quality of the various barley varieties. Five varieties grown in eight different states in the malting barley area were studied comparatively in the laboratory. In addition, some twenty-five less common varieties and hybrid selections grown in one or more locations were tested. Studies were made also of barley samples representing three varieties obtained from farmers fields in the important barley areas of Wisconsin. Briefly summarized, the Oderbrucker, Manchuria, and Velvet varieties yielded somewhat better quality malts over the entire range of growing conditions than any of the other varieties. Ped. 38 was slightly lower in extract, low in water soluble nitrogen in the wort and diastatic power and slightly higher in malt recovery. Trebi in general was high in extract and malt recovery, but very low in water soluble nitrogen in the wort and low in diastatic power. See Figure 8. Some samples of each of the

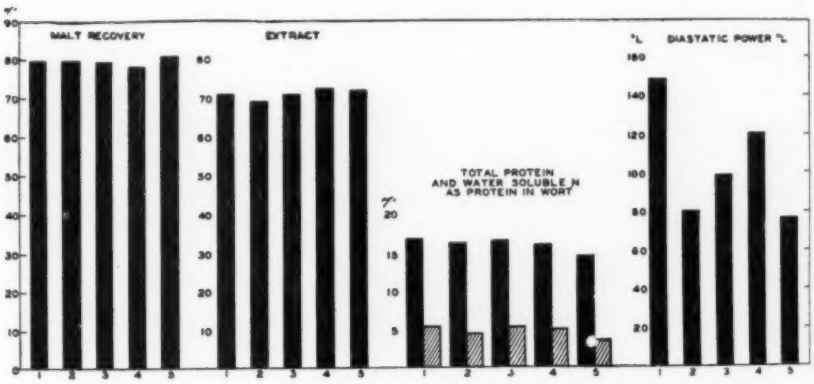

Fig. 8. Graph showing the comparison of the five barley varieties grown in the regional series in 1934. (1) Oderbrucker (Wis. Ped. 5–1), (2) Wisconsin Barbless (Wis. Ped. 38), (3) Velvet (Minn. 447), (4) Manchuria (N.D. 2121), (5) Trebi.

four earlier mentioned varieties were outstanding in quality when grown in particular locations. Some of the less known and newer varieties showed possibilities in malting quality based on the limited tests in a relatively few locations.

The investigations have shown a difference in varietal response to treatment in experimental malting. While it has been generally recognized, that the location in which the barley was grown influenced not only the general quality of the barley, but also the response to malting procedure, little attention has been paid to the varietal reaction in malting response. In rate of water absorption for example each

variety of barley maintains its relative varietal position regardless of the influence of the region in which the barley was grown. This has been observed in other responses and suggests the importance of keeping grain of varieties separate as well as grain from different regions.

Information regarding the agronomic data, disease reactions and malting quality can be summarized briefly in a general statement. The varieties having the highest malting quality are deficient in yield and disease resistance, while the more productive varieties are less suitable in malting quality. This does not necessarily mean there is a correlation between high yield and less suitable malting quality, but rather points to the fact that the newer, high yielding, disease resistant varieties were developed with little or no attention to malting quality.

What is to be our policy and procedure looking to the future? Are we to abandon the program for barley improvement and return to the old type standard malting barley varieties with their recognized deficiencies in production or are we, including the industries, going to combine our efforts in securing the varieties suited alike to profitable production as well as suitable malting and brewing quality? We feel that the latter is the only safe and wise plan and will therefore proceed to the discussion of methods of producing and testing new barley varieties on this basis of carrying the investigation to completion.

The development of new barley varieties with desirable malting quality, high productivity, other suitable agronomic characters and resistance to disease involves the coordinated effort and merits the support of all interested in this crop.

The Development of New Malting Varieties of Barley

The process involves the practical application of the known principles of analysis and synthesis to the development of new varieties. Briefly outlined the method of procedure followed in this program consists in securing suitable parental material, combining desirable characters through hybridization and selecting the promising segregates. In practice this program involves a further survey of the desirable barley germ plasm for hybridizing, the production of the hybrids and intensive selection within large populations of the segregating generations from the hybrids in the search for new combinations of character followed by testing and evaluation.

Parental material has been accumulated and studied during the past few years. Such varieties as Peatland, Korsbyg, Ped. 38, to name only a few, have combinations of certain desirable agronomic characters and resistance to one or more diseases. Oderbrucker,

selections of Manchuria, Peatland, and others are suggested as being desirable parents for transmitting malting quality. Certain of these varieties have been used in the production of hybrids; the segregates of which will soon be available for testing. Other hybrids combining desirable characters will be made during the present season.

Hybridization is a means of combining the characters of two or more parents in a new variety. The genetic factors governing the inheritance of these characters are assorted and appear in new combinations in the hybrid progenies. Intensive selection in large populations of the early segregating, subsequent generations enables the breeder to pick out the individual lines possessing the most desirable combination of characters. Back crossing or further crossing with other desirable parental material makes it possible to intensify certain characters or add still other desirable characters. This process is followed by isolation and stabilization of the new lines. The larger the number of characters to be combined in the new variety, the greater is the task of the plant breeder.

The selecting and testing of these lines involves finding and eliminating the undesirable and keeping the desirable. Selections are made in the earlier segregating generations in individual plant lines principally on the basis of the more obvious agronomic characters including in a gross way yielding capacity. Such characters as kernel type and color, tightness of hull, stiffness of straw, smooth awns, etc., can be determined on relatively few plants. The superior or more promising selections are then subjected to disease tests by producing artificial epidemics of smut, stripe, and scab. The resistant individuals and lines are selected out from the susceptible ones. The malting quality studies are then made on the selected lines at the same time the agronomist is determining the yielding capacity and general agronomic suitability. These final evaluation studies must continue over sufficient range of seasonal conditions and regional environments to determine the adaptation limits and average quality of the new selections.

These intensive studies must be followed by further and more general testing in the various sections of the barley producing areas. Here likewise it is highly important to test for malting quality so that the malting and brewing characters may be well established before the new variety is distributed for commercial production and utilization.

What measurements are to be used in evaluating malting quality in the early selections from segregating generations as well as the barley varieties? In the earlier stages of selection sufficient grain is

not available to secure adequate malting tests; therefore kernel character including endosperm texture, tightness of hull, color and texture of hull, outer coverings of the kernel, etc. must be used as the the important criterion. Both practical experience and scientific investigation suggest there are important kernel characteristics which are associated with malting quality. For example, the friable, mellow type of endosperm is determined in part by the variety regardless of the environment.

Preliminary tests on water absorption and the influence of steeping on kernel character and germination are made on relatively small samples. There are marked varietal differences in these reactions. The more desirable malting varieties show a more rapid rate of water absorption with subsequent rapidity in the modification of the endosperm mass. The tolerance to over-steeping as indicated by uniformity, vigor, and percentage germination varies with the varieties and can be determined early in the selection period.

Malting of small samples gives a preliminary indication of some aspects of quality. The physical determinations, especially uniformity and rate of growth, endosperm modification and kernel weight are important factors in estimating quality. While preliminary determinations on recovery of malt from the small samples of barley are possible, it will probably be necessary to use larger samples to obtain accurate data on these characters. Yield of extract, the amount and distribution of nitrogenous compounds and diastatic power are important factors in determining quality. However, the data from the preliminary studies made thus far indicate that these determinations cannot be used as a final evaluation of malting quality.

The determination of the various physical and chemical factors mentioned lend themselves to more or less routine procedure. Other less tangible factors of quality and stability of various nitrogenous compounds and their relation to yeast nutrition and the condition of the finished product need considerable research; but probably cannot be determined empirically on small samples of grain. Likewise there is need of investigation of the endosperm carbohydrates not measured in the determination of extract, especially in their relation to satisfactory processing in brewing and effect upon quality. And finally there are a series of intangible quality factors perhaps related to general character of the malt such as; aroma, flavor, and similar requirements which seemingly are determined in part by the nature of the barley and in part by the processing. At present, these characters cannot be measured on a satisfactory quantitative basis.

The procedure as outlined involves not only a large amount of

routine testing, but also research in determining other quality factors. While the routine testing is important in evaluating malting factors, fundamental research on other phases of quality must progress with knowledge of the problem. A balance of these two aspects of the program with adequate facilities for testing the selections developed by the plant breeders and comparing these tests with the practical experience of maltsters and brewers in the industries and industrial laboratories is anticipated in the program outlined.

MALTING AND MASHING

HUGO W. ROHDE

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(Read at the Annual Meeting, June 1935)

Under "malting" we understand that process in which grain undergoes a germination, changing the ingredients in such a way as to utilize them in a subsequent process known as "mashing."

Malting is no longer a speculative, rule of the thumb procedure; it is an art as well as a science. During the past ten years great advances have been made in this field; scientific investigations have been applied to practical production, and while the subject is far from exhaustive, many obscure phenomena have been clarified. Deep-seated changes occur during malting and mashing, chiefly insoluble, non-diffusible substances are transformed into simpler bodies. While most grains, such as corn, barley, oats, rye, and wheat can be malted, for our purpose only barley comes into consideration. Certain types of barley are given preference over others. The primary object of malting is to form enzymes, a group of substances formed in the living cell and destined to perform certain physiological changes without they themselves undergoing an alteration. To produce these, no other technical method is known today other than by artificial germination. A definite quantity of enzymes are required, no more and no less, hence our processes must be regulated accordingly to achieve these ends. Cross-section drawings of barley kernels are shown as Figures 1 and 2.

Malting Operation

Malting comprises several operations, namely, the steeping of the barley in water, the growing or the germination of the barley, and finally the drying of the malt; the entire process requiring approximately twelve days. Freshly harvested barley usually germinates poorly. It is therefore necessary to store or age the barley four to six weeks; during this time the barley loses its vegetative moisture, and undergoes and after-ripening. The barley is received at the elevator where it is weighed and sampled. Then it undergoes a cleaning process in which foreign seeds, broken kernels, and impurities are removed. It is then graded into two or three different sized kernels. Barley contains from 85 to 90% dry substance and 10 to 15% moisture. The

Fig. 1. Longitudinal section of a barley kernel, which consists essentially of two parts—the larger part, or the endosperm, which is starch bearing, and the smaller part, the embryo or germ. The outer portion or protecting layer is the spelt. Next to this layer of cells is the pericarp and the testa, and the aleurone layer of cubical cells, the so-called protein layer. The major portion, the endosperm proper, consists of starch cells.

The dividing layer between the embryo and the endosperm is the scutellum or absorbing organ which has important physiological functions. Here the enzymes are formed. The upper part of this organ consists of verticle absorption cells, which take up substances from the endosperm and nourish the embryo.

The embryo contains the rudimentary rootlets, 4 to 6 in number, and the pluma or acrospire which forms the shoot or plant, and which grows as a thin sheath along the wall of the kernel.

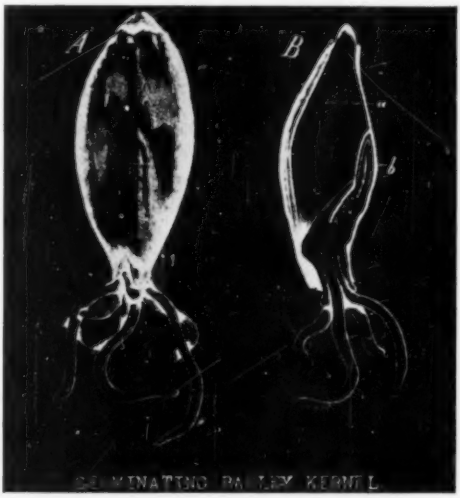

Fig. 2. *A* illustrates a germinating barley kernel having 6 rootlets. *B* is a cross-section of the kernel. The acrospire "*b*" has grown half-way the length of the kernel. Line "*a*" shows the distance the enzymes have been active.

dry substance consists of organic nitrogen-free substance, organic nitrogenous substance, enzymes, vitamins, and mineral matter.

Steeping the Barley

In order to induce germination, additional water is necessary. The cleaned barley is conveyed to the malt-house and dumped into large iron tanks funnel-shaped at the bottom and partly filled with cold, clean water. Here it is again cleaned and washed in running water, air being forced through it thereby causing a continuous circulation. Water enters the kernels mainly at the base or germ end and diffuses into the interior. The light kernels present are skimmed off from the surface. After 45 to 65 hours of steeping, depending upon the type of barley and the temperature of the water, the barley has absorbed sufficient water to start upon the most important step in malting, namely, the growing or germination. The barley now contains from 45 to 48% of water or four bushels of barley will be equivalent to five bushels of steeped barley. In the steeping, the barley not only takes up the requisite quantity of water, but it is washed and aerated so as to give oxygen to it and to remove the carbonic acid formed. The coloring matter and the bitter substances from the husk are extracted and germination is stimulated. To produce this more intensely, the barley is sometimes transferred to another tank.

Germinating the Barley

Germination may take place on a floor, a system now obsolete, in drums, or in compartments. This is a physiological process and in general does not differ when done artificially in the malt-house or in the soil. The essentials are moisture, temperature, and oxygen, just as the life of each individual is associated with these factors. The most favorable temperatures for the initial growth of the barley kernel are 55 to 56° F.; temperatures below 40° F. retard growth and high temperatures above 80° F. change the interior of the kernel too rapidly.

Types of Germinators

The malting drum illustrated in Figures 3 and 4 is so constructed as to permit the barley to germinate under the most favorable conditions. It consists of a closed, slow rotating cylinder provided with air ducts along the periphery and a large discharge air duct in the center. Moist air, properly humidified and temperated, is drawn through the intake air duct, then passes through the loosely packed barley and is discharged through the middle duct and finally leaves the drum on the opposite end. The drums hold the contents of a steep

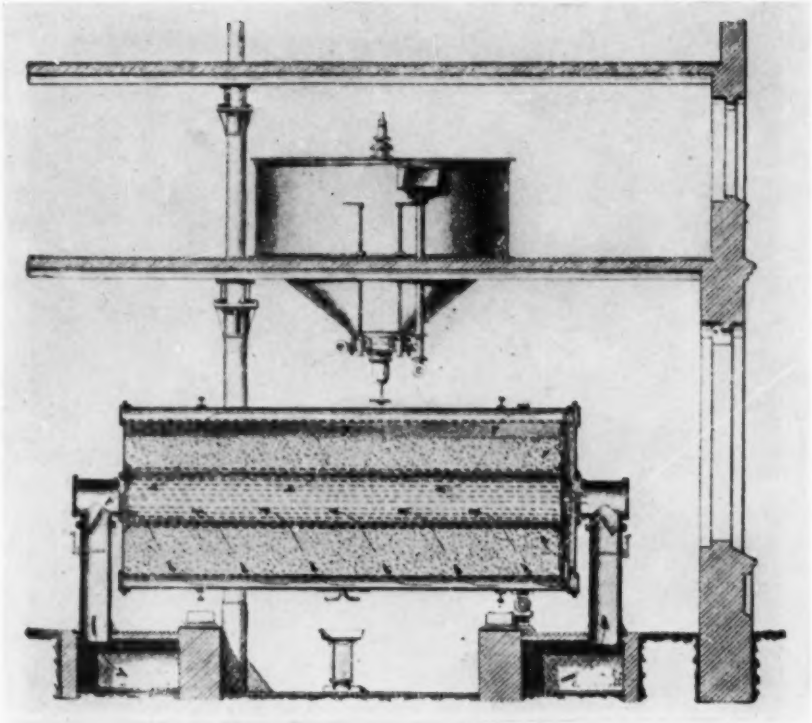

Fig. 3. Malting drum with super-imposed steep tank, having a capacity of 650 bushels.

tank or up to 650 bushels, and make one revolution in about fifty minutes. After a number of hours, the barley begins to show life; it begins to breathe. At the embryo or germ end of the kernel, a white point appears which developes into four to six rootlets. On the inside of the kernel a thin, narrow sheath is formed which grows towards the opposite end. As this continues, moist, cool air is drawn through the germinating grain, which removes the carbonic acid formed in respiration and prevents the temperature from rising too rapidly. The rise in temperature is about 3° to 4° F. each 24 hours beginning at about 55° F. and rising until the temperatures of 70° to 75° F. are reached. Evaporation of moisture also takes place and this water is replaced after 48 hours of germination by sprinkling water from a perforated pipe on the barley. The moisture content should be kept constant during the germinating period.

In the compartment system of malting, illustrated in Figure 5, the barley germinates in long compartments holding up to 5,000 bushels of grain and having a perforated bottom. An agitator slowly moves along the entire length and again returns, gently stirring up the

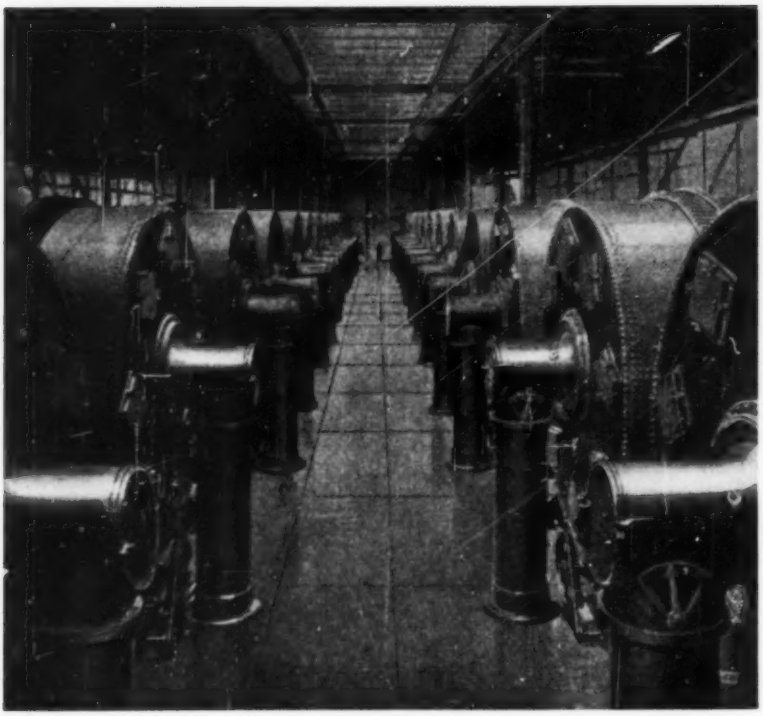

Fig. 4. A battery of malting drums.

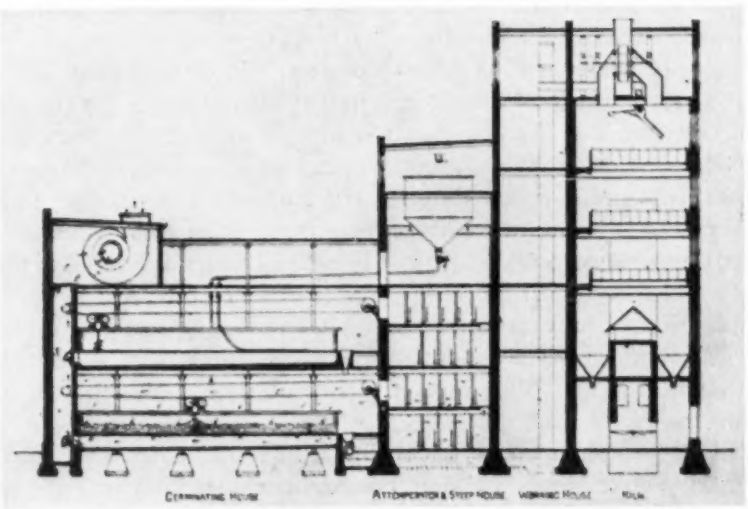

Fig. 5. Diagram of compartment malting system with steep tank and kiln.

barley. In one type of this system, moist air is drawn from above through the grain and is discharged through a duct located below the compartment.

Changes Taking Place in the Barley Kernel During Malting

The changes occurring in the kernel may be divided into a period of growth and into a later period of dissolution of the mealy portion, although both changes take place simultaneously to some degree.

Now let us consider the changes occurring in the interior of the kernel more in detail. Starch as we know is formed from carbonic acid and water under the influence of sunlight and the chlorophyl or green coloring matter in plants and it is stored up as a reserve food material. The starch granules of barley, when viewed with a microscope under high power, are found to be of two kinds, the round, lens-like granules, 10 to 30 μ in size, and the smaller granules, more or less round, and 6 to 12 μ in size. These granules consist of concentric layers which are visible due to a variation of moisture content. Starch or amylum consists of about 98% of pure starch and 2% of protein, fat, cellulose, and mineral matter. The cell wall of the granules consist of amylopectin or starch cellulose, identical with glycogen or animal starch. Amylose of pure starch is a white powder, its suspension in water will not gelatinize or form a starch paste, but a colloidal starch-sol. The amylo-pectin or the cell-wall forming substance is a gummy material which swells up on heating to a colloidal, gelatinous solution socalled starch-gel. It forms a brownish-violet coloration with iodine solution. Starch in its totality consists of a colloidal, non-gelatinizing amylose as sol and a gelatinizable amylo-pectin as gel. In the cold, amylose separates on standing when in water. Upon gelatinizing starch, amylo-pectin forms a buffer thereby preventing the amylose from separating. During gelatinizing the volume of starch may increase a hundred fold. The blue coloration with iodine is not a chemical reaction, but an adsorption reaction (a solid solution of iodine in starch) the coloration of which will disappear after some time.

Barley contains other nitrogen-free substances but only in small quantity. Next to starch the protein compounds are of most importance for our purpose.

Vitamins are also present in barley in small amounts.

Barley Most Suited for Malting

In the United States we are mostly concerned with the 6-rowed barley growing in the middle states. These barleys contain from 11 to 14.5% of protein. We require a relatively high protein barley because we use unmalted cereals in the mashing process. It is not possible to

classify the protein substances along chemical lines because we lack knowledge of their exact structure. For simplicity we divide proteins into three groups, namely:

1. Simple proteins as albumins, globulins, protamins and albuminoids
2. Compound proteins
3. Protein transformation products

It will lead too far to discuss these.

Roll of Enzymes in Malting and Mashing

One group of substances, no doubt the most important in malting and mashing, constitute the enzymes. The chemical nature and structure of these peculiar substances is still unknown. They are formed in the living cell, perform certain functions, are destitute of life and cannot reproduce. We group them among the catalysts, *i.e.*, substances which even in very small quantity will speed up chemical reactions without being used up themselves. They differ from the inorganic catalysts in that they are specific in their reactions. One particular enzyme will perform only a specific function, the starch transforming diastase will act upon starch only and not on maltose. The influence of temperature is of importance, each enzyme having an optimum temperature at which it is most active. The reaction of the substrate is also well marked.

Enzymes are most likely formed in the epithelial cells of the scutellum. The largest amount of enzymatic substances are near the embryo where the soluble proteins will assist in their formation. As germination continues, the enzyme cytase dissolves the hemicellulose wall bounding the starch granules or at least makes the wall pervious to the enzyme diastase or amylase as it is now called. Even though amylase does not reach its height of importance in malting, its presence will be of interest. It is not distributed uniformly throughout the endosperm or mealy portion of the kernel. It is assumed that the action of amylase on starch occurs in several steps. Perhaps the so-called transformation amylase of ungerminated barley and the secretion amylase of germinating barley are identical, the latter being associated with more of the enzyme amylophosphatase. Supplementing these amylases, there appears evidence of a coamylase, an activator, which seems to attack the starch when the ordinary amylase is no longer active.

Under high magnification of the microscope, this action of amylase on starch can be followed very beautifully. The starch granules will form depressions on the surface, the concentric layers will separate,

radial crevices will form and the enzymatic action will continue until all of the material is consumed.

The ripe germ is free from starch. During the period of germ formation, starch is formed from saccharose, only to be converted back into saccharose which is assimilated and serves to form tissues. This particular form of starch is called transitory starch or temporary starch.

The enzymes of protein transformation can be divided into subgroups, the proteases degregating the albumin to peptones and polypeptids, the peptidases splitting the polypeptids into amino acids, while the amidases break down the amino acids to ammonia. This does not exhaust the number of enzymes of germinating barley, but it suffices to show some of the complex changes taking place during the malting process.

Drying the Green Malt

After six to seven days, germination being normally conducted, the acrospire or plumule has grown about three-quarters of the length of the kernel. The green malt having attained its proper dissolution now contains about 45% of moisture. In this condition it is likely to undergo unfavorable changes, hence the moisture must be reduced to about 4 to 5%. This operation, the drying or kilning, brings about physical and chemical changes, comprising loss of moisture, reduction in volume, weight, color, aroma, and taste. The kilning takes place in two phases, the initial drying or withering and the final drying or kilning. This must be cautiously performed or the resulting malt will be flinty and lack the desired aroma. The kiln consists of 2 or 3 perforated floors, one above the other, a heating element at the bottom and a ventilating arrangement. The green malt is spread upon the upper floor and after 24 hours this floor is tilted and the malt falls on the floor below where the drying is continued, requiring 48 to 72 hours as the case may be. Hot air is drawn through the malt until finally a temperature of about 160° F. is reached which is held for three hours to produce light colored malt. The malt is now taken from the kiln and it is cleaned, the rootlets or malt sprouts are removed and the malt is stored several months before use. During this storage the malt becomes more mellow.

Malts of high diastatic properties are usually made from small kerneled barley and kilned at relatively low temperatures so as not to unduly injure their diastatic power.

A new departure of kilning malt has recently been introduced, namely, drying under a high vacuum of 29 inches. After the green malt has withered for a time, it is placed into drums and heated by

water circulating through pipes. Two drums are connected with a unit. In the first drum the moisture is reduced to 12% at temperatures of 75° to 110° F., and the kilning is completed in the finishing drum at 110° to 150° F. These drums rotate one-half revolution each way at a slow speed. The water-vapor drawn from the malt is condensed.

Mashing Operation

The *mashing operation* is the continuation of the solution of the malt. Here not only are the soluble substances extracted from the malt, but also substances soluble in water at elevated temperatures are formed. This process is termed "mashing" and the solution obtained is called "wort."

The malt is crushed between rollers so as not to injure the spelt or husk for this will serve as a filtering medium. It is then dropped into a closed metallic cylindrical vessel (see Fig. 6) provided with an agitator

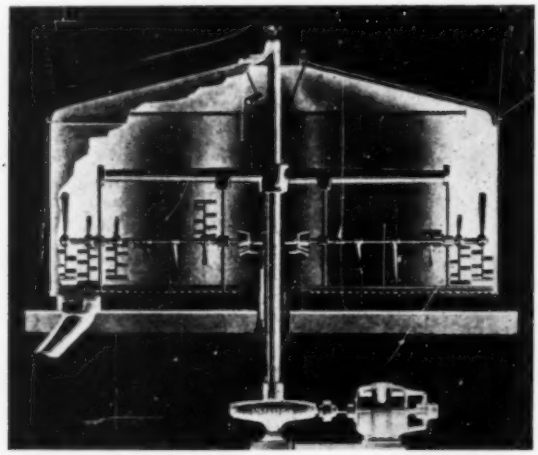

Fig. 6. Cross-section of a cylindrical mash-tun showing stirrer, perforated bottom, and water sparging pipe.

and a perforated or slitted false bottom, also a pipe for spraying water and connections for cold water, hot water, and steam. Water of about 120° F. is put into the mash-tun, a weighed quantity of malt is added and the mixture thoroughly mixed. The mineral salts of the water play an important role in the mashing, as some are actually harmful. Soft waters must be hardened, and too hard waters must be dehardened. The enzymes formed during malting become active, each enzyme performing its specific function and having its optimum at certain strictly defined temperatures. The time element is also of importance as well as the acidity expressed as the hydrogen-ion concentration or the pH value, the most favorable being 5 to 5.5. The

acidity may be natural or by the addition of acid-forming bacteria or by adding lactic or other acids.

During mashing, starch which is present in largest quantity in malt, is acted upon by the enzyme amylase forming dextrines and maltose sugar. The intermediate substances are collectively known as dextrines and are characterized by their behavior with polarized light, Fehling's Solution, solubility in alcohol of different concentrations, with yeasts of various types and with iodine solution. Of the better known dextrines we have amylo-dextrine very much resembling starch, and the erythro-dextrines which give a reddish coloration with iodine. The achroo-dextrines which do not react with iodine and of which three have been found, have been identified as malto-dextrines. The final product of the action of diastase on starch is maltose or malt sugar. Under favorable conditions, there is formed 80% of maltose and 20% of dextrines during the mashing process. This ratio may be shifted by proper manipulation of concentration, temperature, time, and acidity. The most favorable temperatures for maltose formation are 135° to 143.5° F. and a pH of 5 to 5.5. The more concentrated the diastase, the more maltose being formed.

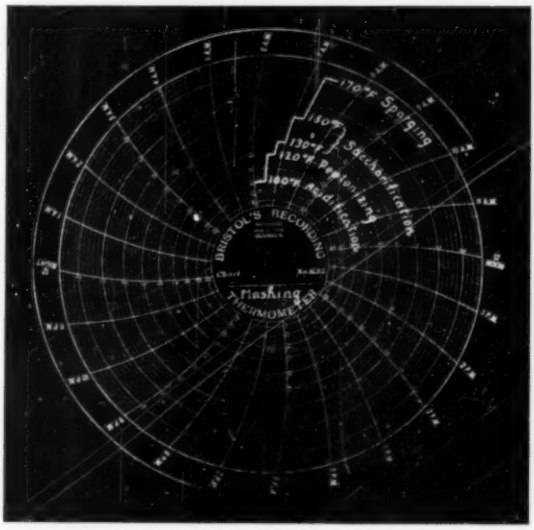

Fig. 7. Mash-tun temperature chart, showing at which temperatures the different enzymes are active. At the lower temperature, 100° F., acidification of the mash occurs. At 120° F. and above, the proteolytic (Peptonizing) enzymes are active. Saccharification falls between the ranges of 130° to 150° F. and finally at 170° F. we have the mashing-off temperature.

Besides the important action of amylase on starch, we have other enzymes breaking down the complex protein molecule, although the quantity of these substances is far smaller. Here we obtain substances

which are more complicated, the protein transformation being less clearly understood. The optimum temperature of activity of the different proteolytic enzymes varies but is around 118° to 122° F. and a pH of 4.3 to 5.

The temperatures in mashing are raised to about 175° F. with hot water and steam or with a boiling cooker-mash which consists of gelatinized corn products or rice (see Fig. 7).

After completing the mashing, the insoluble material will settle on the bottom and serve as a filtering medium. The clear wort is now drawn off, the residues washed with hot water removing the adhering extract. The remaining spent grains are dried in a drum drier (see Fig. 8) and constitute a nutritious cattle and horse feed.

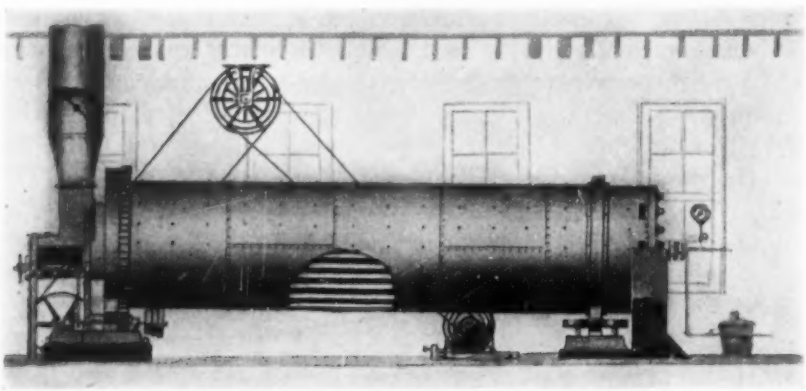

Fig. 8. Grain drier for drying the spent grains not utilized in mashing. The grains are first pressed to remove water whereupon they pass through the drier consisting of a rotating device with steam pipes. The grains enter one end of the drum and are discharged at the other end carrying about 7 to 8% of moisture.

The method of mashing described above is known as the infusion mashing method which is universally used in the United States. There are modifications of various kinds to meet the conditions of mechanical equipment, material, and product desired.

In the above, a brief attempt was made to describe the malting and mashing processes and the changes which accompany these. While the barley is subjected to physical and chemical fluctuations beyond the control of man, it becomes necessary to modify certain operations to meet the changing conditions. Here, as with many other processes, involving organized materials, laboratory control is most essential.

THE DETERMINATION OF MOISTURE IN BARLEY MALT.

A Comparison of Methods and Apparatus for this Purpose [1]

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U. S. Department of Agriculture, Washington, D. C.

(Read at the Annual Meeting, June 1935)

Introduction

The problem of rapid and accurate determination of moisture in cereals and cereal products is continuously before the cereal chemist. Rapid moisture testing procedures are desirable in order to shorten the routine of making moisture determinations and accurate moisture determinations must be the rule because of the intimate relationship existing between the moisture content of a commodity and its intrinsic value, keeping quality, and transportability.

Members of this Association in the past, have carried out extensive studies for the purpose of developing methods of procedure and apparatus for determining the moisture content of wheat and wheat products, and, after extensive collaborative trials, this Association has adopted these methods as official methods.

The importance of a rapid, yet accurate, determination of the moisture content of barley malt and malt adjuncts is of as great importance to the user of these products as it is to the user of other cereals and cereal products. This can be readily seen if one considers the relationship existing between the moisture content of malt and the "extract yield." A difference of 1% in the moisture content of the malt can change the percentage of "extract yield" by approximately 1% of the "extract yield" percentage. For example, assume an "extract yield" figure of 70% and a moisture content of 4%; converted to a dry-matter basis the "extract yield" figure would be 72.91%. On the other hand, if the moisture content of the malt was 5%, the "extract yield" figure would be 73.68%, a difference of 0.78%.

One of the many activities of the newly appointed Committee on the Standardization of Methods for Brewing and Malting Control was to give consideration to a study of the methods in present day use in malt and brewing laboratories for the analysis of malt. Information

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and data were sought with respect to the manner in which moisture determinations on malt were made. To acquire this information samples of malt were sent to 20-odd laboratories connected with brewing and malting institutions with a request that moisture determinations be made on the malt samples. The results obtained by the collaborators together with a description of the apparatus and procedures used in obtaining the moisture content results were requested.

The moisture content results submitted by the various analysts, as well as the answers to the questionnaire regarding the methods and apparatus used in obtaining the moisture content figures, were amazing. Differences in moisture content results were found to be as much as 2.5%. Among the 18 laboratories submitting moisture content results 15 different moisture determination procedures were found to be in use. These procedures varied with respect to (1) the size of sample taken for the moisture determinations, (2) the type of oven used for drying purposes, (3) the duration of the drying time, and (4) the temperature used for drying. The size of the malt sample used for moisture determinations varied from 2 to 10 g.

The types of drying ovens used were far from uniform. While the electrically-heated ovens were most commonly used, ovens heated by gas and even by steam were also reported. In addition the vacuum oven operated at 25 mm. pressure, at the temperature of boiling water, (98–100° C.) was used by two analysts.

The time of drying varied from 10 minutes to 5 hours, the majority of analysts drying the malt sample for 3 hours. A few analysts reported returning the sample to the drying oven for an additional hour's drying.

The temperature of drying varied from 98° C. to 132.5° C. Most analysts, however, heated their drying ovens over the range from 102° to 105° C.; the most frequently used temperature being 103° C. to 104° C.

With such an accumulation of contradictory data relating to the making of moisture determinations before the committee, it was decided to undertake an extensive research with respect to the determination of moisture in malt and to study, in so far as it was possible, the merits of as many of the methods for making moisture determinations as were found during the survey in order to recommend to the Association a standard procedure for the determination of the moisture content of malt. It was decided, also, to recommend such alternate moisture testing procedures as could be used to obtain results equivalent with those obtained by the standard method.

Experimental

Methods Used in Making the Moisture Determination Studies

Ten methods for determining the moisture content of malt were used in this study. They were:

A. The 130° C. air-oven method (See Cereal Laboratory Methods, page 29, section 2*b*).
B. The Bidwell-Sterling method (See Cereal Laboratory Methods, page 9, section 3).
C. The method embodying the use of the Carter-Simon oven (See Cereal Chemistry, 3: 419).
D. The vacuum desiccator method. Official A. O. A. C. procedure (See Official and Tentative Methods of Analysis of the Association of Official Agricultural Chemists, page 278, section 3, par. 5 and 6) modified by the use of anhydrous P_2O_5 instead of moisture-free H_2SO_4.
E. The standard vacuum-oven procedure (See Cereal Laboratory Methods, page 29, section 2*a*).
F. The standard method of the Malt Analysis Standardization Committee (See Official Methods for the Analysis of Malt, published by the Malt Analysis Standardization Committee, Chicago, 1935).
G. The vacuum-oven method using a pressure of 25 mm. of mercury but heating the samples for 18 continuous hours at a temperature of 70° C.
H. The water-oven method, *i.e.*, heating the malt sample for 5 hours without the aid of a vacuum at the boiling point of water.
I. The Brown-Duvel method (See U. S. Department of Agriculture Bulletin 1375 revised, or Cereal Laboratory Methods, page 3, section 4*a*).
J. The dielectric procedure using a Cambridge dielectrically operated moisture meter.

Brief statements follow with respect to the technique of operation for the various above listed methods.

The moisture boxes used were the low-form aluminum type with tightly fitting friction lids, having a diameter of about 55 mm. and height about 15 mm.

An approximate 3-g. sample (sample was taken on a catch-weight basis) of the finely ground malt was used in all of the determinations made by all of the methods.

The thermometers used in the drying ovens were calibrated for immersion and so placed in the ovens that their mercury bulbs were within a few centimeters of the top of the moisture boxes.

In the vacuum ovens (not vacuum desiccators) the moisture box covers rested lightly on the top of the moisture boxes. In all other ovens the moisture box tops were removed from the moisture boxes.

The 130° C. air-oven method was used exactly as is common for the determination of moisture in flour and wheat.

In the preliminary trials with the Bidwell-Sterling method the procedure described in "Cereal Laboratory Methods," page 9, section 3, was tried. It was early observed, however, that cork joints were entirely unsatisfactory and that ground glass joints were necessary to obtain accurate results. It was found after investigation also that

50 g. of finely ground malt were necessary to furnish sufficient water to permit of an accurate reading; also that 150 cc. of toluene were necessary as scorching frequently occurred when using only 75 to 100 cc. of toluene.

The apparatus finally adopted, therefore, consisted of a 300 cc. Erlenmeyer flask provided with ground-glass necks for connection with a Mohr pipette. The Mohr pipette in turn was provided with ground-glass joints for connection with a Liebig condenser. All other procedures with respect to this method were as described on page 9, section 3 of Cereal Laboratory Methods, with the exception, of course, that 150 cc. of toluene were used instead of 100 cc.

While the first series of moisture tests by this method was unsatisfactory, subsequent research indicated the method had possibilities as a rapid alternate method and considerable research was expended on the method in order to make it produce moisture content results closely equivalent to the standard method. A description of the research necessary to accomplish this equivalency will be given later.

The Carter-Simon oven method is the so-called "travelling oven" type of moisture tester. With this apparatus the samples are inserted into the heating chamber in 5-minute intervals, 15 minutes being required for a single sample to pass through the heating chamber. The oven was operated at a temperature of 132.5° C.

The vacuum desiccator method is that recommended by the A. O. A. C. (See Official and Tentative Methods of Analysis of the Association of Official Agricultural Chemists, Third Ed., page 278, section III, par. 5 and 6) modified only in substituting anhydrous P_2O_5 for moisture-free H_2SO_4. The reason for this substitution is that phosphorous pentoxide is a much stronger dehydrating agent than sulphuric acid. Further, sulphuric acid when used as a desiccant must be specially prepared. This is a troublesome and tedious procedure. In addition, when the moisture determination is nearing completion a constant weight is not as easily obtained in a sulphuric acid desiccator as in one filled with anhydrous P_2O_5.

It was found that 30 days were required to complete a moisture determination by the vacuum desiccator procedure. This being the case it is hardly necessary to make first weighings before the 18th day, with additional weighings at the end of the 24th and 30th days. If, in any instance, the percentage of moisture was found to be greater than 0.1% on succeeding weighings, the boxes were returned to the desiccator for an additional 6 days. Moisture determinations were called complete when after continued exposure to P_2O_5 the moisture content of the sample did not increase by .05%.

To insure success of this method, the surface or crust of the P_2O_5 must be broken before the samples for moisture test purposes are placed in the desiccator. The desiccator must also be well sealed (with desiccator wax or grease) before the air is expelled. It is a good policy to test the desiccator the following day for leaks that may have occurred during the night. Evacuation of the air in the desiccator was carried to a pressure of 10 mm. of mercury or less. The desiccators were stored at room temperature.

It is reasonable to assume that the moisture content results obtained by this method more nearly represent the true moisture content of malt than the moisture results by any other method largely because of the absence of the action of heat on the sugars in malt.

The vacuum-oven method was undertaken exactly as is official for determining the moisture content of flour or feeds, namely, heating the samples of malt at a temperature of 99 to 100° C. under a pressure of 25 mm. of mercury for a period of 5 hours. No subsequent weighings were made.

The vacuum-oven method operated at 70° C. is a modification of the method described above. The technique, however, of carrying out the moisture determination was the same as described above, except for the temperature of drying, 70° C., and the time of heating.

The official method of the Malt Analysis Standardization Committee (Chicago) is essentially the Congress method and has been previously described in the Salzburg Agreement for Commercial Malt Analysis. Inasmuch as this method has not found general distribution in the American literature, it is quoted verbatim from the handbook "Official Methods for Analysis of Malt" (Malt Analysis Standardization Committee (Chicago, 1935)):

"The moisture oven should be provided with automatic temperature control designed to keep the temperature within 0.5° C.

"The oven shall be of a size large enough to accommodate all the samples on one shelf, and in such a manner that no sample is outside the area indicated by test to give comparable results in duplicate samples.

"The oven should be standardized by placing the weighed duplicate samples in the oven at 103° to 104° C. and drying for 3 hours. They are then weighed and redried for 1 hour longer. If the moisture content has increased by more than 0.1% the temperature of the oven is raised 1° C. and the oven standardized with a new sample of malt. Thus the oven is to be standardized at the lowest temperature below 106° C., which will give a moisture content that after 3 hours of drying will be within 0.1% of the value attainable at the same temperature within 4 hours.

"The weighing bottle may be of glass or aluminum, provided with a tight fitting cover and have a diameter of approximately 40 mm. for a 5 gram sample, or 55 mm. for a 10 gram sample.

"The ventilators of the oven shall be open during the entire drying period and the door is on no account to be opened during the three hours of drying.

"The sample should be of the same fineness as the finely ground malt for the determination of extract.

"Weigh approximately 5 grams of whole malt and grind through a clean dry mill directly into a weighing bottle (a larger quantity, *i.e.*, 10 grams may be used,

provided the weighing bottle is of correspondingly large diameter so as to provide a sample of equal depth in the drying dish). Thereafter brush all malt in mill into dish and cover dish immediately with tight fitting cover.

"After the weight of the sample has been ascertained, using the analytical balance, it is placed in the oven previously heated to the drying temperature, cover of dish is removed and heated for exactly three hours at the temperature which has been determined by the preliminary test. At the end of this time the weighing bottle, with the cover replaced, is transferred to a desiccator and weighed on the analytical balance as soon as cold.

"The moisture is reported with one decimal to the nearest tenth per cent."

Briefly interpreted, this method calls first for standardization of the heat of the drying oven. This is accomplished by placing duplicate samples of malt into the drying oven at a temperature of 103°–104° C., and drying the malt sample for 3 hours. The samples are then removed from the oven, cooled and weighed, and then returned to the oven for an additional hour's drying. If the moisture content of the malt increases by over 0.1% due to this additional hour's heating, the temperature of the oven is raised 1° C. and the heat of the oven is again standardized with new samples of the same malt. However, the maximum oven temperature is not to exceed 106° C. Thus the oven is to be standardized at the lowest temperature below 106° C. which will give a moisture content after 3 hours of drying within 0.1% of the value attainable at the same temperature with 4 hours drying.

The electric oven used in this study was found to be in standard heating condition when operated at 104°–105° C. under load. When empty the oven temperature rose to 106.5° C.

When using the Brown-Duvel method, 100 g. of unground malt were immersed in 200 cc. of oil. The "cut-off" temperature was 168° C. All other technique such as standardizing the moisture tester, cooling time, reading of graduates, etc., was carried out as described in U. S. Department of Agriculture Bulletin 1375 revised.

In the dielectric method 150 g. of unground malt were placed in the condenser of the moisture meter and readings taken. Meter readings were plotted against moisture readings and a conversion chart prepared. The accuracy of the instrument was determined as it predicted moisture contents as shown by the chart.

Material Used

The malts used in this investigation consisted of choice, fancy, and standard grades of brewers' malts obtained from domestic and foreign sources as well as samples of black malt, caramel malt, coffee malt, dextrine malt, distillers' malt, and hi-dried malt. It will be seen later that the moisture contents of the malts used in this study varied from 3.0 to 9.8%.

Preparation of Sample

Each sample of malt was ground to a fine state of subdivision by means of a Labconco mill, type RF, set at XF. Care was exercised during the grinding of the malt so that a sample of uniform moisture content was obtained. To assist in this effort the ground malts were sealed and set aside for several days to insure moisture equilibrium.

At this point in the investigation the type of mill used to grind the malt is not of importance provided, of course, a fine grist is obtained, because the investigation at this point concerns itself with moisture determinations on the same sample of malt by different methods and procedures and is not directed toward ascertaining the absolute moisture content of specific samples of malt. Later in the investigation the influence of grinding apparatus on malt moisture determination results will be discussed.

Variance between Replicate Moisture Determinations by Different Moisture Determination Methods

The variance between replicates is of outstanding importance in connection with the evaluation of a method. Whenever material and time permitted each moisture test method was studied for variations in moisture results between replicates. For this purpose five replicate moisture determinations were made by methods *A*, *C*, *D*, *E*, *F*, and *G*, while for methods *B* and *I*, three replicates and two replicates respectively were made. In the instance of methods *B* and *I*, the reduced number of replicates was occasioned by the large size of sample (50 and 100 g. respectively) necessary to complete the tests.

The maximum range in moisture determination results between replicates by the various methods now being used for making moisture determinations is shown in Table I. The extent of the variation between replicates is not constantly the same for any given method. This is to be expected when it is considered that all malts are not of the same composition, varying as they do in the quantity and kind of sugars and other simplified carbohydrates present. However, on the basis of averages, there is a very definite trend for some methods to show greater variances between replicates than others. From Table I it is clear that better agreements between replicates can be consistently expected from methods *D*, *E*, and *G* for all types of malts, while for methods *A*, *B*, *C*, and *F* greater variances between replicates are to be expected with any given malt, and with some malts variances will be greater than with others. For example, with method *A*, whereas the average deviation between replicates was of the order 0.15%, the maximum and minimum deviation was from 0.26% to 0.04%. For

TABLE I

SHOWING MAXIMUM DEVIATION BETWEEN FIVE REPLICATE MOISTURE DETERMINATIONS ON VARIOUS MALTS BY 7 DIFFERENT METHODS

Sample number	Method						
	A [1]	*B* [2]	*C* [3]	*D* [4]	*E* [5]	*F* [6]	*G* [7]
	%	%	%	%	%	%	%
1	0.14	0.1	0.11	0.06	0.05	0.12	0.06
2	0.22	0.2	0.14	0.06	0.08	0.16	0.07
3	0.26	0.3	0.15	0.10	0.15	0.13	0.04
4	0.20	0.1	0.29	0.07	0.02	0.13	0.05
5	0.12	0.2	0.24	0.03	0.04	0.13	0.04
6	0.14	0.2	0.25	0.03	0.07	0.14	0.07
7	0.18	0.2	0.20	0.06	0.02	0.16	0.05
8	0.21	0.0	0.21	0.06	0.07	0.25	0.06
9	0.15	0.2	0.21	—	0.04	0.14	0.08
10	0.12	0.0	0.04	—	0.03	0.18	—
11	0.21	0.4	0.18	—	0.03	0.17	—
12	0.24	0.2	0.18	—	0.06	0.14	—
13	0.09	0.0	0.26	—	0.02	0.14	—
14	0.19	0.4	0.12	—	0.02	0.11	—
15	0.14	0.0	0.21	—	0.05	0.14	—
16	0.16	0.2	0.16	—	0.06	0.12	—
17	0.24	0.1	0.10	—	0.08	0.14	—
18	0.09	0.2	0.09	—	0.10	0.17	—
19	0.17	0.0	0.13	—	0.12	0.10	—
20	0.09	0.2	0.06	—	0.04	0.19	—
21	0.04	0.2	0.14	—	0.19	0.19	—
22	0.12	0.2	0.10	—	0.09	0.14	—
23	0.01	0.0	0.10	—	0.09	0.20	—
Average	0.15	0.16	0.16	0.06	0.07	0.15	0.06
Maximum	0.26	0.4	0.29	0.10	0.19	0.25	0.08
Minimum	0.04	0.0	0.04	0.03	0.02	0.10	0.04

[1] *A*—130° C. air-oven method.
[2] *B*—Bidwell-Sterling method.
[3] *C*—Carter-Simon method.
[4] *D*—Vacuum-desiccator method.
[5] *E*—Vacuum-oven method at 100° C. for 5 hrs.
[6] *F*—M.A.S.C. official method.
[7] *G*—Vacuum-oven method at 70° C. for 18 hrs.

method *B*, although the average deviation for all samples tested was 0.16%, the maximum deviation was 0.4%, the minimum deviation 0.0%. This same phenomenon also holds for method *C*. With methods *E*, *F*, and *G* not only is the average variation between replicates of a smaller order, but the maximum and minimum variation in replicates for all samples was of a low order.

From an examination of the data in Table I it is reasonable to expect duplicate moisture determinations to agree within 0.2% when using methods *A*, *B*, *C*, and *F*; within 0.1% by methods *E* and *G*; and by 0.05% by method *D*. The data in Table I indicate that the most consistent moisture test results can be expected between the various

methods in the following named order: *G, D, E, F, A, C B.* It also appears from the data shown in Table I that the percentage of error may be high when using methods *A* and *C*, within the limit of accuracy when using methods *B*, *E*, and *F*, and that the error is of small magnitude when using methods *D* and *G*.

Choice of a Standard Method for Determining the Moisture Content of Malt

While a specific method may give ideal replicate determinations it may also indicate moisture results which are inaccurate in so far as moisture in cereal products is generally understood.

Moisture in a commodity exists in at least three forms—water of constitution, water of crystallization, and absorbed or adsorbed water. This last type of water is also frequently referred to as hygroscopic water. By appropriate means hygroscopic water may be removed or may be conceived of as having been removed from a cereal or cereal product without changing the chemical composition of the product, the only physical change taking place being that due to desiccation resulting from the removal of all of the uncombined water. What is meant by moisture, therefore, is the water which may be added to or taken from a product without altering its chemical composition, and does not therefore include water of constitution or water of crystallization.

The ideal method therefore for the determination of moisture in malt is the one which removes all the hygroscopic water and disturbs to the least extent water of crystallization or water of constitution. The removal of even hygroscopic water in malt is attended with difficulty, especially by methods which resort to the application of heat because of the very labile condition of the malt sample, a condition brought about as the result of its high content of the more simple carbohydrates (sugars).

The melting point of some of the sugars frequently found in malt are: fructose, 94°–97° C.; maltose, 118° C.; glucose, 146° C.; and sucrose, 160°–170° C. The caramelization of these sugars begins very close to their melting point, especially if the temperature is prolonged for any length of time. Accompanying the caramelization phenomena decomposition products form, which, of course, introduce errors when moisture determinations are being made.

In determining upon a method or procedure which would fulfill conditions with respect to the removal of all of the hygroscopic water, and disturb to the least extent the water of crystallization and constitution in the malt sample, and one which would be free of the application of heat so that caramelization and other decomposition effects

TABLE II

MOISTURE CONTENT OF MALT AS DETERMINED BY DIFFERENT OVEN METHODS COMPARED WITH THE MOISTURE RESULTS OBTAINED BY THE STANDARD METHOD (VACUUM-DESICCATOR METHOD)

Sample number	Moisture determination method						
	D [1]	*A* [2]	*C* [3]	*E* [4]	*F* [5]	*G* [6]	*H* [7]
	%	%	%	%	%	%	%
1 [8]	5.18	5.87	5.78	5.49	5.22	5.09	4.75
2	5.81	6.91	6.50	6.24	5.85	5.61	5.63
3	5.36	5.96	6.15	5.48	5.35	5.20	5.30
4	5.16	5.93	5.73	5.36	5.19	5.30	5.36
5	4.63	5.36	5.10	4.95	4.72	4.70	4.88
6	6.49	7.27	6.95	7.02	6.61	6.40	6.08
7	6.39	7.18	6.93	6.85	6.50	6.50	6.12
8	6.67	7.32	7.18	7.03	6.75	6.67	6.22
9	6.42	7.04	6.91	6.85	6.46	6.39	6.12
10	6.35	7.02	6.84	6.72	6.41	6.35	5.95
11	6.63	7.46	7.28	7.04	6.79	6.80	6.45
12	7.43	7.63	7.46	7.59	7.24	7.05	6.72
13	7.07	7.59	7.50	7.45	7.19	6.94	6.55
14	7.42	7.70	7.63	7.62	7.35	6.99	6.64
15	6.80	7.58	7.40	7.08	6.81	6.80	6.47
16	7.00	7.78	7.60	7.32	7.00	6.98	6.65
17	8.63	9.38	9.01	8.93	8.60	8.55	8.09
18	8.70	9.28	9.14	9.02	8.64	8.60	8.12
19	8.86	9.30	9.24	9.04	8.75	8.70	8.29
20	8.71	9.48	9.17	8.93	8.51	8.50	8.27
21	8.08	8.67	8.49	8.23	7.98	7.88	7.57
22	7.58	8.05	8.00	7.68	7.39	7.36	7.12
23	5.65	6.02	5.96	5.76	5.60	5.37	5.00
24	5.72	6.03	5.96	5.82	5.62	5.51	5.07
25	5.43	5.57	5.63	5.49	5.24	5.16	4.68
26	9.81	10.34	10.41	10.25	9.99	9.73	9.40
27	6.05	6.66	6.38	5.95	5.86	5.81	5.67
28	7.37	8.15	8.03	7.66	7.46	7.32	7.28
29	4.98	5.53	5.64	5.17	4.84	4.88	4.85
30	4.39	5.21	4.87	4.48	4.23	4.21	4.32
31	4.34	5.07	4.87	4.58	4.23	4.24	4.34
32	4.75	5.38	5.38	4.85	4.63	4.73	4.72
33	4.66	5.51	5.28	4.93	4.53	4.54	4.54
34	5.62	6.54	6.30	5.84	5.54	5.61	5.68
35	5.34	5.93	5.82	5.54	5.30	5.19	5.26
36	5.24	5.73	5.65	5.04	4.93	4.99	4.86
37	4.81	5.46	5.44	4.90	4.68	4.63	4.62
38	4.53	4.75	4.96	4.83	4.50	4.40	4.21
39	4.84	5.68	5.54	5.00	4.74	4.62	4.90
40	4.41	5.11	4.92	4.68	4.28	4.33	4.53
41	4.20	5.08	4.97	4.61	4.13	4.08	4.87
42	4.44	5.14	4.87	4.50	4.40	4.40	4.58
43	4.29	4.78	4.69	4.49	4.36	4.46	4.38
44	5.20	6.22	6.10	5.77	5.26	5.21	5.67
45	5.37	5.63	5.65	5.58	5.41	5.60	5.05
46	9.18	9.56	9.53	9.45	9.20	9.03	8.74
47	4.14	4.58	4.61	4.37	4.01	4.08	3.82
48	4.09	4.46	4.60	4.32	4.05	3.90	3.85
49	5.65	6.37	6.15	5.95	5.59	5.66	5.31
50	6.35	7.26	7.20	6.76	6.41	6.34	6.10
Average	6.04	6.67	6.55	6.30	6.04	5.94	5.79
Deviation from *D*		+.64	+.51	+.26	.00	−.10	−.25

TABLE II—(*Continued*)

Sample number	Moisture determination method						
	D [1]	*A* [2]	*C* [3]	*E* [4]	*F* [5]	*G* [6]	*H* [7]
	%	%	%	%	%	%	%
51 [9]	7.10	7.88	7.61	7.37	7.09	6.91	6.60
52 [10]	5.48	5.89	5.72	5.59	5.31	5.45	5.02
53 [11]	3.63	3.84	3.60	3.75	3.41	3.32	3.27
54 [12]	5.45	5.91	5.83	5.65	5.46	5.31	5.21
55 [12]	3.59	4.04	4.00	3.77	3.48	3.59	3.75
56 [13]	4.75	4.86	4.81	4.83	4.72	4.63	4.40
57 [13]	3.51	3.78	3.71	3.65	3.53	3.68	3.38
58 [14]	4.34	4.50	4.53	4.55	4.41	4.46	4.16
59 [14]	3.09	2.98	3.00	3.05	3.01	3.15	2.82

[1] *D*—Standard vacuum-desiccator method.
[2] *A*—130° C. air-oven method.
[3] *C*—Carter-Simon method.
[4] *E*—Vacuum-oven method at 100° C. for 5 hrs.
[5] *F*—M.A.S.C. official method.
[6] *G*—Vacuum-oven method at 70°C. for 18 hrs.
[7] *H*—Water-oven method.
[8] Samples 1 to 50 were brewers or distillers malts.
[9] Rye malt.
[10] Hi-dried malt.
[11] Dextrine malt.
[12] Caramel malt.
[13] Coffee malt.
[14] Black malt.

would not be important, the vacuum desiccator method of determining moisture was decided upon as the standard or referee method.

This decision was reached after a considerable number of trials were made in which it was conclusively shown that no detectable caramelization of the malt sample took place when using this procedure. Further, previous experiences using the vacuum-desiccator method for the determination of moisture in ground cereals and cereal products had shown that the moisture test results obtainable by the vacuum-desiccator method were the equivalent of those obtained by the 100° C. 5-hour 25 mm. pressure vacuum-oven method or the 130° C. air-oven method, in which methods the balance existing for the removal of hygroscopic moisture, as distinguished from water of crystallization and water of constitution, is apparently satisfactory. Further, from the data recorded in Table I it is apparent that highly satisfactory replicate determinations are possible by this method and that the sample effect is at a minimum.

Comparative Moisture Contents of 59 Malts as Determined by Different Methods of Making Moisture Determinations

Oven Methods

In order to compare the relative moisture determining efficiency of some of the oven methods found to be in use for determining the moisture content of malt, moisture determinations were made on 59 different lots of malt by the methods listed in Table II.

Observations with Respect to the Accuracy and Efficiency of the Various Oven Methods

130° C. air-oven method—(*A*): Compared with the standard method, this method gave consistently high moisture content results. Extensive discoloration of the malt samples took place with this method. Prolongation of heating at the temperature recommended for this method causes further discoloration of the malt samples and gives even higher moisture content values.

Carter-Simon oven method—(*C*): The moisture results obtained by this method were slightly less than those obtained by Method *A*, but were considerably higher than those obtained by the standard Method (*D*). Discoloration of the malt was almost as great as accompanied the use of Method *A*. Modification of this method so as to give lower moisture values would be somewhat difficult on account of the triple influence of the factors—time, temperature, and weight of sample. Surprisingly, also, the moisture content of the sample has a great influence on the efficiency of this method.

The Carter-Simon apparatus has certain weaknesses. In the first place the thermoregulator is not as sensitive as it should be. Usually the make and break of the relay lag behind the actual heat generated by the oven causing a temperature variable practically impossible to overcome. With the oven adjusted to the standard 132.5° C. temperature, the introduction of samples of malt of variable weight and moisture content so influenced the temperature of the heating chamber that the specified heating conditions rarely prevailed. For example, if samples of malt of reasonably wide variances in moisture content followed one another through the heating chamber, a sample of high moisture would cool the heating chamber and the temperature of drying for the other malt samples in the chamber would not be correct because the chamber would not regain its proper temperature before the time elapsed for heating the other malts had passed. It is good practice therefore, if this type of oven and moisture test procedure is to be used to start the moisture determinations (after the temperature of the oven has reached 135° C.) with 3 blanks or samples which are discarded after passing through the oven, adjusting the oven chamber to 132.5° C. while the blanks are passing through the oven.

Vacuum-oven method at 100° C.—(*E*): This method gave uniformly consistent moisture values—0.3% higher than those obtained by the standard method. Variance between replicates was small. However, discoloration of the malts due no doubt to the 5-hour heating at 100° C., was quite noticeable.

Vacuum-oven method at 70° C.—(*G*): With this method the time of

heating was increased and the temperature of the heating chamber decreased obviously for the reason that it was desired to use a temperature where decomposition of sugars due to heat would be at a minimum. Trials were made by this method using heating intervals of 6, 12, and 18 hours. Six and 12 hours of heating at 70° C. produced low moisture results in comparison with the results obtained by the standard method. Increasing the time interval to 18 hours increased the moisture content slightly, but the moisture results were still slightly under the results obtainable with the same samples by the standard method. Slight increases in moisture content, yet below the standard method results, were obtained by increasing the time of heating to 24 hours. However, it appeared that heating could be prolonged indefinitely with a small but gradual increase in moisture content values. However, accompanying the heating time, discoloration of the sample ("caramelization") was more pronounced, a phenomenon indicative of decomposition processes.

Malt Standardization Committee Method—(*F*): Moisture results by this method checked those by the standard or vacuum desiccator method very closely.

From the appearance of the heated malt samples caramelization appears to be very slight.

Under the conditions surrounding the study of this method, *i.e.*, heating of the sample for 3 hours at 104°–105° C., 3 hours heating produced the same moisture results as obtained by heating for 4 hours. An important feature in standardizing the drying oven for this moisture determination method is to bear in mind that the temperature of the oven must be at the maximum of the set range practically all of the time the drying is taking place. A lowering in oven drying temperatures of 1° C. will influence the moisture test results surprisingly. However, it was found that if the oven is standardized as recommended by the Malt Analysis Standardization Committee, using malts which have been checked for moisture content by the vacuum-desiccator method, moisture results can be obtained by this method which agree within 0.1% with the vacuum-desiccator method.

Water-oven method—(*H*): The moisture values obtained by this method were in most instances low in comparison with the vacuum desiccator or standard method. Agreement between replicates was excellent. Discoloration of the sample due to heating was not very noticeable. Results by this procedure should approximate those obtainable by the standard method of the Institute of Brewing (British), as the description of their standard method for the determination of moisture in pale malts as given on page 518 of Volume 39 of the Journal of the Institute of Brewing is the same in all respects as the

procedure followed under Method *H* with the exception of the time of heating. Method *H* calls for a 5-hour heating rather than the 3-hour heating interval specified by the Institute of Brewing.

Distillation Methods

Comparative Accuracy of the Bidwell-Sterling Method and the Brown-Duvel Method for the Determination of Moisture in Malt

The efficiency of two distillation methods for determining the moisture content of malt was studied, these were the Bidwell-Sterling method and the Brown-Duvel method.

Both methods were tried with numerous modifications to perfect them for use as rapid methods for the analysis of moisture in malt with the results shown in Table III. From an examination of the

TABLE III

Moisture Content of Malts as Determined by Distillation Procedure, *i.e.*, Bidwell-Sterling and Brown-Duvel Methods, Compared to the Results by Method *D*

Sample number	Method			Sample number	Method			Sample number	Method		
	D[1]	*B*[2]	*I*[3]		*D*[1]	*B*[2]	*I*[3]		*D*[1]	*B*[2]	*I*[3]
	%	%	%		%	%	%		%	%	%
1[4]	5.18	5.20	5.10	21	8.08	8.30	7.30	41	4.20	4.20	4.10
2	5.81	5.90	5.85	22	7.58	7.40	7.20	42	4.44	4.40	4.20
3	5.36	5.40	5.15	23	5.65	5.30	—	43	4.29	4.40	4.20
4	5.16	5.20	5.20	24	5.72	5.40	—	44	5.20	5.30	5.15
5	4.63	4.60	5.05	25	5.43	5.00	—	45	5.37	5.40	5.10
6	6.49	6.50	6.30	26	9.81	10.00	—	46	9.18	9.00	9.00
7	6.39	6.50	6.40	27	6.05	5.60	5.80	47	4.14	3.80	3.60
8	6.67	6.70	6.35	28	7.37	7.30	7.60	48	4.09	4.00	4.10
9	6.42	6.40	5.85	29	4.98	4.80	4.80	49	5.65	5.60	5.50
10	6.35	6.40	6.25	30	4.39	4.20	4.20	50	6.35	6.30	6.40
11	6.63	6.70	6.30	31	4.34	4.00	4.40				
12	7.43	7.20	7.00	32	4.75	4.60	4.60	Average	6.04	5.96	5.87
13	7.07	7.00	7.50	33	4.66	4.50	—				
14	7.42	7.40	7.15	34	5.62	5.60	6.00	Devia-			
15	6.80	6.70	6.55	35	5.34	5.20	5.40	tion			
16	7.00	7.00	6.90	36	5.24	5.00	5.10	from *D*		−.08	−.13[5]
17	8.63	8.50	8.30	37	4.81	4.70	5.00				
18	8.70	8.60	8.65	38	4.53	4.40	4.40				
19	8.86	8.70	8.85	39	4.84	4.80	4.30	51[6]	7.10	7.00	—
20	8.71	8.60	8.50	40	4.41	4.40	4.05	52[7]	5.48	5.30	5.4
								53[8]	3.63	3.20	3.0
								54[9]	5.45	5.20	—
								55[9]	3.59	3.50	3.2
								56[10]	4.75	4.40	—
								57[10]	3.51	3.50	—
								58[11]	4.34	4.40	—
								59[11]	3.09	3.00	—

[1] *D*—Standard vacuum-desiccator method.
[2] *B*—Bidwell-Sterling method.
[3] *I*—Brown-Duvel method.
[4] Samples 1 to 50 were brewers or distillers malt.
[5] Average based on 45 tests.
[6] Rye malt.
[7] Hi-dried malt.
[8] Dextrine malt.
[9] Caramel malt.
[10] Coffee malt.
[11] Black malt.

data given in Table III it will be seen that the Bidwell-Sterling method operated as will be described, produced results which agreed remarkably well with those obtained by the standard vacuum-desiccator method. On the other hand, the moisture results produced by the Brown-Duvel method were quite erratic. With some malt samples a close agreement of moisture content values were obtained between the results obtained by the Brown-Duvel method and the results obtained by the vacuum-desiccator method, while with other samples of malt the comparative moisture results were very divergent.

It is clear from the data recorded in Table III that the Bidwell-Sterling method was the more reliable of the two distillation methods.

Selection of Alternate Methods for Determining the Moisture Content of Malt

Drying Oven Methods

It can be seen from the data in Table II that the moisture content of the various lots of malt listed is dependent upon the method of determination used. The several methods used divide themselves into (1) those which constantly produce high moisture content results when compared to the standard vacuum desiccator method, and (2) those which produce slightly low moisture test results when compared to the standard method, and (3) one method which gives results practically of the same order as the standard vacuum desiccator procedure.

Methods *A* (130° C. air-oven method), *C* (Carter-Simon method), and *E* (vacuum-oven method at 100° C. for 5 hrs.) produce high results, Methods *G* (vacuum-oven method at 70° C. for 18 hrs.) and *H* (water-oven method) low results, while the results obtained from Method *F* (M. A. S. C. official method) are in excellent agreement with the standard vacuum desiccator procedure (*D*). Moisture determination results by the various methods in comparison with method F are charted in Figures 1, 2, 3, and 4.

When using methods *A*, *C*, and *E* marked evidences of caramelization were present. Caramelization of the sugars as well as decomposition products of amino acids, no doubt are responsible in measure for the higher moisture results recorded by these methods. This is confirmed in part by the following experiment.

Water extracts of several of the malts were compared before and after drying by the different oven methods with the results shown in Table IV.

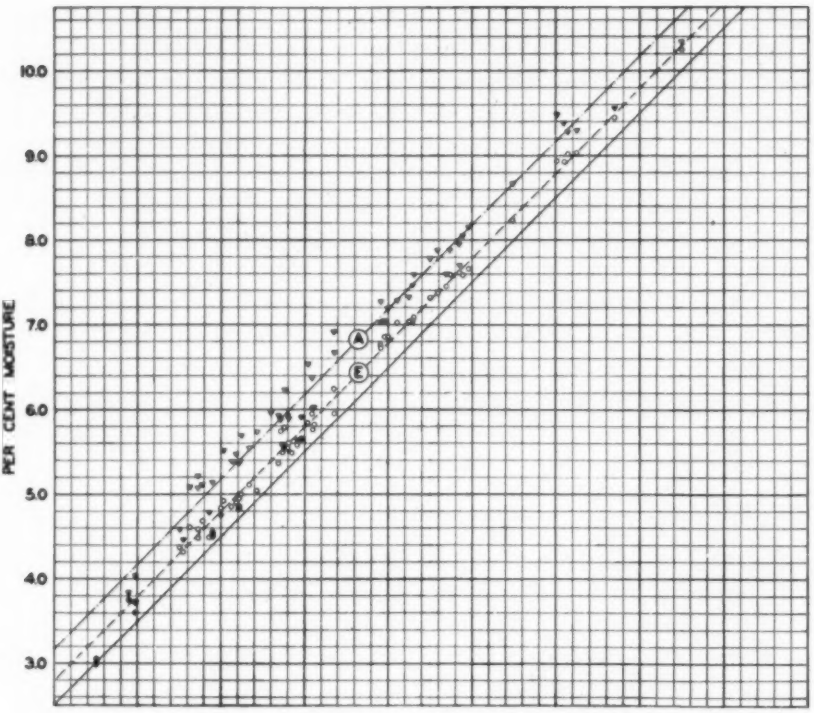

Fig. 1. Comparison of moisture determination results by various methods.

Method *A*, ▽ — · — · — · —
Method *E*, ○ ----------
Method *F* ——————

TABLE IV

DISCOLORATION OF BARLEY MALTS DURING DRYING BY VARIOUS OVEN METHODS

Method	Time	Temperature	Discoloration
D—Standard vacuum-desiccator	28 days	Room	None
G—Vacuum-oven—70° C.	18 hours	70° C.	None
F—M. A. S. C. official	3 hours	104°–105° C.	Very little
E—Vacuum-oven—100° C.	5 hours	100° C.	Little
C—Carter-Simon	15 minutes	132.5° C.	High
A—130° C. air-oven	1 hour	130° C.	Very high
Original sample	None	None	None

Since caramelization represents a loss in weight due to breakdown of sugars, and is not therefore unbound or hygroscopic water, it is obvious that any method involving extensive caramelization of the malt is not suited for the determination of moisture in malt, and conversely, it is obvious that any method which involves the minimum of heat and heating time with a minimum of caramelization should

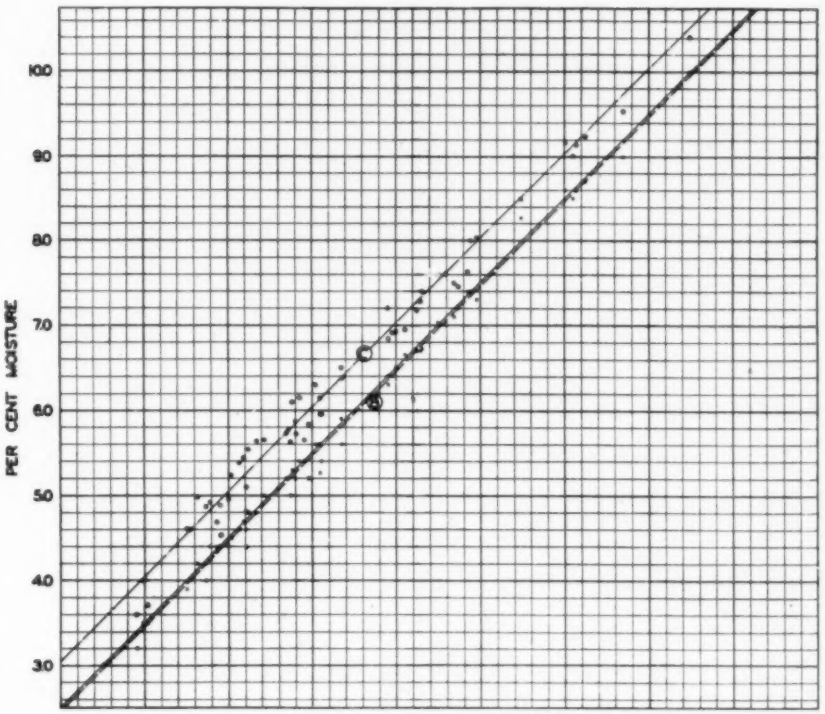

Fig. 2. Comparison of moisture determination results by various methods.

Method *C*, ⊘ — · — · — ·
Method *B*, × ----------
Method *F* ——————

produce moisture results closest to the correct moisture content. This eliminates from the group of oven methods capable of expressing moisture content, Methods *A*, *C*, and *E*, and brings to the foreground Methods *F* and *G*.

As method *G* gives consistently low results in comparison with the standard vacuum desiccator procedure, and also because water-oven operation is extremely difficult to standardize due to variations in the boiling point of water as a function of altitude, it can also be eliminated from consideration, even though good replicates can be expected with the method and the results obtained by its use are about 0.2% below the vacuum desiccator standard method.

This leaves the 103°–104° C. air-oven method sponsored by the Malt Analysis Standardization Committee (Chicago) as the only suitable alternate method among the six drying-oven methods studied.

Further trials with this oven method were therefore undertaken to determine its efficiency with respect to a number of factors.

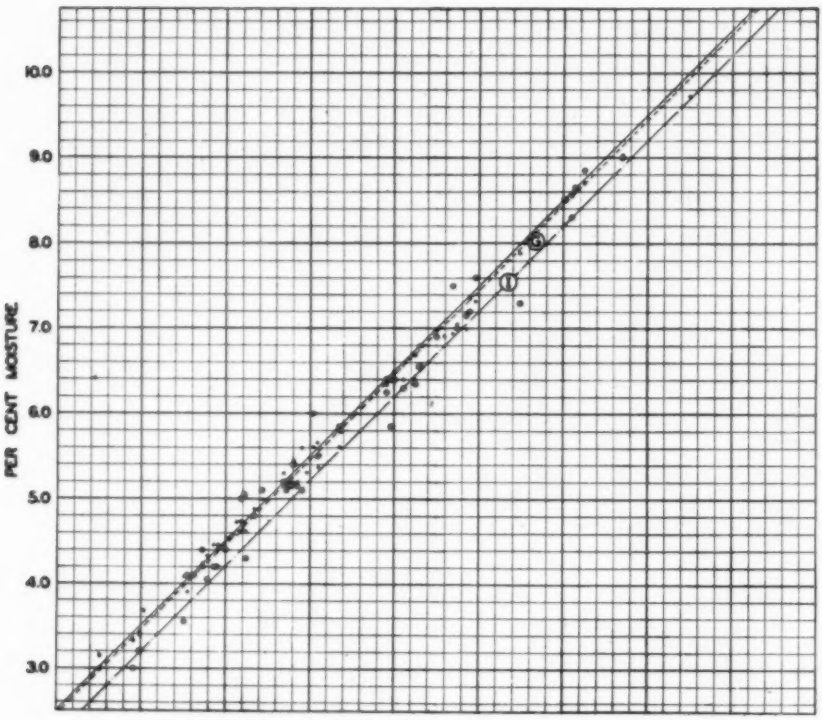

Fig. 3. Comparison of moisture determination results by various methods.

Method *G*, + ----------
Method *I*, ⊘ — · — · — ·
Method *F* ————————

Studies were made with respect to the daily variance in moisture determinations on the same samples of malt by the 103°–104° C. air-oven method. (See Table V.)

TABLE V

DAILY VARIATION IN MOISTURE DETERMINATIONS BY THE 103°–104° C. AIR-OVEN METHOD USING THE SAME GROUND SAMPLE OF MALT

	Sample					
Day	1	2	3	4	5	6
	%	%	%	%	%	%
1	6.16 [1]	6.62	8.58	7.57	4.21	8.70
2	6.19	6.66	8.61	7.66	4.35	8.85
3	6.35	6.74	8.71	7.59	4.38	8.87
4	6.32	6.73	8.66	7.70	4.42	8.89
Average	6.25	6.69	8.64	7.63	4.34	8.83
Maximum range	.19	.12	.13	.23	.21	.19
Average variance		0.18				

[1] Average of duplicate determinations.

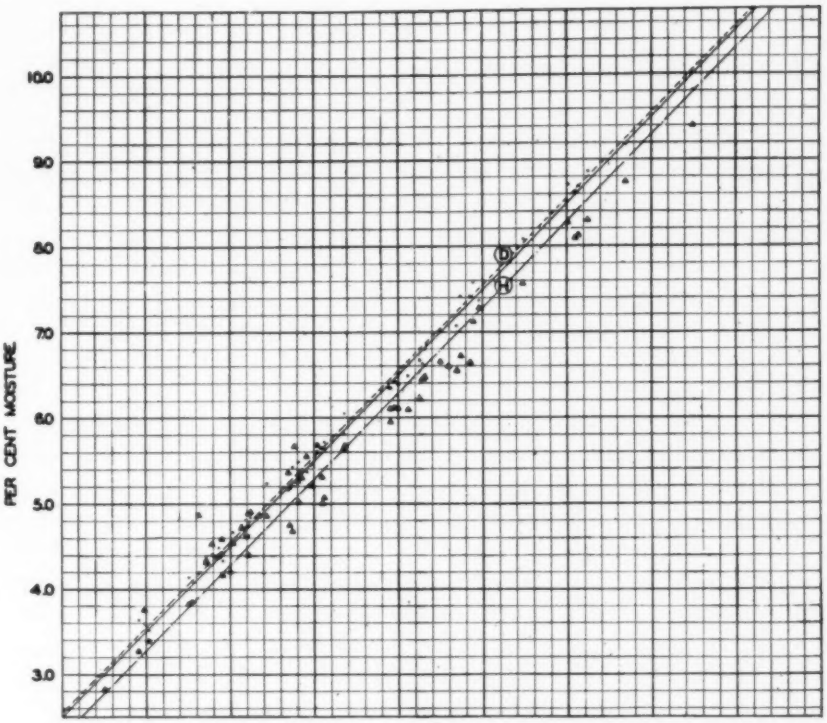

Fig. 4. Comparison of moisture determination results by various method.

Method *D*, ● ----------
Method *H*, Δ — · — · — ·
Method *F* ————————

The data shown in Table V demonstrate that an average daily variance of approximately 0.2% can be expected when using this method. That this variation is inherent in the method and is not the result of widely divergent duplicates was proven by the fact that the duplicate determinations with each sample over the daily interval agreed as follows:

	Sample					
Day	1	2	3	4	5	6
	%	%	%	%	%	%
1	0.04	0.02	0.04	0.09	0.09	0.01
2	.02	.08	.07	.08	.07	.04
3	.04	.04	.00	.04	.08	.02
4	.05	.00	.04	.09	.02	.03

An Alternate Distillation Method

Recommended Procedure for Using the Bidwell-Sterling Method

In exploring the possibility of using the Bidwell-Sterling Method for the determination of the moisture content of malt it soon became apparent that certain modifications in the published technique and apparatus would be necessary.

Numerous trials demonstrated that apparatus with interchangeable ground-glass connections must be used to gain accurate results. Further, the size of the Erlenmeyer flask must be 300 cc., because of the large amount of sample which has to be used in order to accomplish a moisture yield of from 1.5 to 5 cc.

With a 50-g. sample of finely ground malt it was soon learned that the rate of distillation was affected by the quantity of toluene used and that if the bottoms of the Erlenmeyer flasks did not rest squarely on the surface of the hot plate, slow and irregular distillations resulted.

It was determined that 150 cc. of toluene was the most satisfactory quantity to use. When 100 cc. of toluene or less was used the malt-toluene suspension frequently bumped and the toluene solution discolored rapidly. Scorching frequently occurred with some samples of malt when only 100 cc. of toluene was used. Solutions discoloring but little checked more closely with the standard method.

200 cc. of toluene was not satisfactory because with this amount of boiling solution and a 600-watt hot plate, there was not sufficient heat generated to regulate the rate of distillation; distillation was slow and low moisture values were obtained.

Experiences indicated that the recommended rate of distillation for this method, namely, starting at the rate of 2 drops per second and gradually increasing to 4 drops per minute, was the most satisfactory.

Armed with the above facts an apparatus with ground-glass joints was assembled. 50 g. of ground malt was suspended in 150 cc. of toluene and the suspension distilled for 60 minutes. The distillation started at the rate of 2 drops per second, the rate gradually increasing to 4 drops per second at the end of the 60-minute interval.

To insure uniformity in the distillation rate, it was necessary to have a constant and uniformally applied source of heat. A special oil bath [4] with a false bottom was constructed to accomplish this

[4] The bath used in connection with this method had a capacity of 3½ liters, and was of the following dimensions: Length, 24½ in.; width, 5½ in.; depth, 3 in. inside and 3½ in. outside (the bottom of the bath was flanged ½ in. to prevent it from slipping on the hot plate).

The bath had a false bottom made of wire of ½ in. square mesh. The false bottom was raised ½ in. above the bottom of the bath.

The cover of the bath was 25½ in. by 6 in., with ¾ in. flanges underneath to prevent it from slipping. The holes in the bath cover were 2½ in. in diameter, spaced 4½ in. from center to center, and 3½ in. from center to end on each end.

Three holes were cut in the top of the oil bath to provide space for No. 5 stopper with thermometer so that temperature readings could be taken.

purpose. The false bottom in the bath was provided so that the flasks were raised about ½ inch from the bottom of the oil bath, thus eliminating hot spots. The bath was filled with a medium grade of engine oil and the temperature of the oil bath was raised to 120° C.

Instead of heating the Erlenmeyer flasks on a hot-plate as commonly done, the Erlenmeyer flasks were immersed in the oil bath to such a depth that the surface of the oil was above that of the toluene suspension. Having done this it is necessary to connect the flask with the Mohr pipette and Liebig condenser immediately as toluene boils at 110° C. There is some leeway, however, as distillation does not begin for 3 to 4 minutes. The temperature of the oil bath gradually rises to 140° C. At this temperature rapid distillation takes place. The condensers were washed down with 10 cc. of toluene after 50 minutes of distillation and again after 55 minutes of distillation. At this point the supernatant liquid in the Mohr pipette is practically clear and should be so in *all* cases. The Erlenmeyer flasks were disconnected after 60 minutes of distillation, the sides of the Mohr pipette scraped with a policeman to remove any adhering drops of water and readings made.

Results by Electrical Methods

Dielectric Results

Results by this moisture testing procedure demonstrated that due to a wide variation in the weight of the various malts the mass of a given weight of malt varied considerably. As the result of this condition the filling of the condenser of the moisture meter to a uniform level was impossible. This situation apparently influences the moisture determination results adversely because beyond a moisture content of 5%, the dielectric method produced erratic results. Below the 5% moisture level the moisture determining ability of the apparatus showed a greater degree of accuracy. It is believed, therefore, that as a plant control instrument where exact moisture determinations are not necessary, and where in the malt drying process it would be desirable to know promptly how far drying has proceeded, particularly in relation to the 5% level, the instrument has moisture determining possibilities.

Other Electrical Apparatus

An attempt to use the Tag-Heppenstall moisture meter for grain for the determination of the moisture content of malt was unsuccessful. The Tag-Heppenstall device as now constructed will not record resistances of cereals containing less than 7% of moisture with any degree of accuracy. The majority of our domestic malts usually contain less than 7% of moisture. The instrument therefore does not have any value for the determination of the moisture content of malt.

Other Studies with Respect to Moisture Determinations on Malt

Effect of Grinding and Sampling

The Seck mill has been chosen as the standard grinding apparatus for the purpose of making moisture determinations on malt by the Malt Analysis Standardization Committee (Chicago) and by the Institute of Brewing (British). It is claimed by the first group that the Seck mill is the only one known which can grind a malt sample without loss of moisture.

In order to determine whether other types of grinding apparatus, such as is found in the average cereal chemist's laboratory, would not suit equally as well as a Seck mill for the fine grinding of malt, studies were made with two additional types of grinding equipment.

For this purpose a bushel sample of malt was carefully mixed and quartered and 12 aliquot portions removed for the purpose of making moisture determinations. Each portion contained approximately 150 g. of malt.

50-g. portions of each of the 12 150-g. aliquots were ground on three different types of mills—(1) a Labconco mill set at XF, (2) a Hobart coffee mill set at 1.0, and (3) a Seck mill set at 0.0, at which setting 5% of the sample was retained on a No. 30 U. S. Standard screen.

All moisture determinations were made by the M. A. S. C. Method (*F*).

The results of the study are given in Table VI.

TABLE VI

Influence of Type of Grinding Mill on the Moisture Content of Malt

Aliquot	Labconco mill	Seck mill	Hobart mill
	%	%	%
1	5.67	5.74	— [1]
2	5.75	5.87	5.91
3	5.85	5.99	5.74
4	5.67	6.10	6.15
5	5.89	5.95	6.08
6	6.00	5.83	5.88
7	5.87	5.57	6.10
8	5.66	5.65	5.82
9	6.03	5.82	5.59
10	5.74	5.69	5.85
11	5.79	5.77	5.71
12	6.00	5.90	5.67
Average	5.83	5.82	5.86
Maximum	6.00	6.10	6.15
Minimum	5.66	5.57	5.59
Range	.34	.53	.56

[1] Lost.

It would appear from these data that the Hobart coffee mill is the least suited for the fine grinding of malt, and that the Seck mill is no better suited for this purpose than the Labconco mill, in fact from the data at hand it is not so efficient in preventing moisture losses.

Effect of Size of Sample

The effect of the size of the sample of malt used for moisture determination was also studied. These data have been organized and are presented as Table VII. It would appear from these data that if proper precautions have been taken that the size of the sample taken is of minor consequence in relation to the accuracy of the moisture test result as the moisture test result obtained with a three-gram sample was approximately the same as that obtained with a 10-gram sample. It should be pointed out, however, that the size of the moisture box was sufficient so that the depth of the sample exposed to heat was not excessive.

TABLE VII

Variation Due to Use of 3, 5, and 10 Gram Sample

Weight of sample	Sample number					
	1	2	3	4	5	6
3 g.	6.16	6.62	8.58	7.57	4.21	8.70
5 g.	6.25	6.69	8.70	7.73	4.39	8.92
10 g.	6.14	6.68	8.60	7.61	4.29	8.77

Conclusions

The vacuum desiccator method, *i.e.*, drying of the sample over anhydrous P_2O_5 in a desiccator maintained at a pressure of 10 mm. of mercury or less until a constant loss in weight has been reached, has been selected as the standard or referee method for making moisture determinations on malt because of the absence of caramelization or decomposition phenomena which commonly accompany the oven methods used for moisture determination purposes. In addition, it is believed that this method removes all of the hygroscopic or unbound water and leaves undisturbed water of crystallization or water of constitution.

Compared to the vacuum desiccator method, moisture results obtained by the following air-oven methods gave average moisture values of the following order:

130° C. air-oven method: 0.63% higher than the standard method.

Vacuum oven operated at 25 mm. pressure at 100° C. for five hours: 0.25% higher than the standard method results.

Carter-Simon method: 0.5% higher than standard desiccator method results.

Vacuum oven operated at 25 mm. pressure but at 70° C. for 18 hours: 0.1% lower than the standard method results.

Malt Analysis Standardization Committee method, *i.e.*, heating for 3 hours at a temperature between 103°–104° C.: Results were practically equivalent to the standard method, being 0.04% lower than the standard method results.

Water-oven method: 0.26% lower than the standard method results.

By use of Figures 1, 2, 3, and 4, the moisture results obtained by any of the moisture methods studied can be expressed in terms of the standard M. A. S. C. method or the vacuum desiccator method by locating the moisture content by any given method on the abscissae of its respective moisture line, following the ordinate line for the same value on the standard moisture line and reading back on the abscissa line for the true moisture content.

The standard method of the Malt Analysis Standardization Committee (Chicago) can, therefore, serve as an alternate standard method for the determination of moisture in malt.

When using the Malt Analysis Standardization Committee (Chicago) method for determining moisture (*i.e.*, the 103°–104° C. air-oven method) a daily method variance, equivalent in moisture results of approximately 0.2%, is to be anticipated.

Experiments likewise show that the combined daily variance due to the method plus the sample variance are of the order 0.3%. In other words, an analyst using the Malt Analysis Standardization Committee's method cannot be expected to agree closer than 0.3% when analyzing different portions of the same lot of malt.

The Brown-Duvel method does not consistently determine the moisture content of malt with accuracy.

Comparative trials with two distillation methods demonstrated that the Bidwell-Sterling method with modifications as to apparatus and method of operation can be successfully used as a rapid method for determining the moisture content of malt. This method, on the basis of averages, gave moisture results closely approximating those obtained by the vacuum-desiccator method, if carried out in the following manner and with the following apparatus:

Apparatus—(1) Bidwell-Sterling equipment made with interchangeable ground-glass joints. (2) Oil bath. (3) 600-watt hotplate.

Reagents—C.P. Toluene, B.P. 110° C.

Method—Place 50 g. of finely ground malt in the 300 cc. Erlenmeyer flask of the Bidwell-Sterling apparatus, add 150 cc. of toluene. Immerse the flask and contents in an oil bath which has been previously heated to 120° C., to such a depth that the surface of the oil is above that of the toluene suspension.

Connect the Erlenmeyer with the Mohr pipette at once. The temperature of the oil bath will gradually rise to 140° C. At this temperature rapid distillation begins. Allow distillation to continue for 50 minutes. At this time wash the pipette with 10 cc. of toluene. Allow distillation for 5 additional minutes and wash again. At this point the supernatant liquid in the Mohr pipette should be practically clear in all cases. Disconnect the apparatus after 60 minutes of distillation, scrape the sides of the Mohr pipette with a policeman to remove any adhering drop of water and make readings.

The dielectric method for making moisture determinations has not been developed to that degree of accuracy where reliable moisture determinations can be expected from all types of malts and ranges in moisture content.

The conductivity type of electrical moisture testing apparatus as now constructed will not determine the moisture content of malt.

RECENT DEVELOPMENTS OF EUROPEAN BREWING CHEMISTRY

E. SINGRUEN

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(Read at the Annual Meeting, June 1935)

Upon returning from a two months' trip to Europe, undertaken for the purpose of studying recent scientific developments in the brewing industry, I was asked to briefly outline the problems of this industry in the various European countries and the efforts of the research workers in this field to solve them.

Before going into details, I shall give a brief description of the manner in which commercial malt analysis is handled. After years of co-operative efforts on the part of scientific stations and individual malting and brewing plants, nearly all European laboratories interested in this work are united in the Central European Committee for Standardization of Malt Analysis. On the other hand, because of the

different types of beer produced in England and the changes necessary in brewing methods—top fermentation for the production of ales, stouts and porters—the English institutions have established standard methods for malt analysis which meet their specific requirements. All members of the Central European Committee have agreed upon and are using the standard methods described in the Salzburg Agreement.[1] These methods form the basis of the methods for malt analysis adopted by the malting and brewing industries in this country [2] and by this Association. By using these methods results obtained by different analysts compare very favorably and can easily be duplicated.

My journey first led me to Switzerland. The Swiss brewing industry, although suffering under the general European economical crisis has retained a high standard and maintains a fine control and research laboratory in Zurich. Since Swiss beers are rarely pasteurized their main problem centers around keeping quality of the beer and chillproofness. This necessitates a rigid biological control of the yeast and the elimination of conditions favorable to the growth of micro-organisms in the finished beer. In this connection comprehensive studies have been undertaken by the Swiss Brewing Station and Dr. F. Kutter to control the composition of the wort, particularly in respect to those nitrogenous compounds which may endanger the brilliancy and keeping quality of the finished beer. For this purpose a large series of experiments have been conducted to study the factors influencing "breakage," that is proper coagulation of the undesirable proteins in the kettle and their elimination through precipitation during cooling.

As a contribution of the Swiss Brewing Station to the work of the Central European Committee a series of methods for the determination of the extract in barley have been studied and compared. (Schweizer Brauerei-Rundschau, *46:* 161(1935).)

As the analytical fractionation of the products of proteolysis offers great difficulties and comparatively little is known about the functions of the individual fractions, attention is largely paid to the development of suitable methods for their determination. Formol-titration which serves as an index for the low molecular N compounds, such as the amino-acids, has given valuable results in this respect.

In direct contrast to Swiss brewing practices are those of Denmark, where 98% of all beer brewed is sold as pasteurized bottle beer and a large percentage is shipped to distant foreign countries.

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[1] Copies of the English translation of the Salzburg Agreement may be obtained from the Chairman, Malt Analysis Standardization Committee of the A. A. C. C., Box 161, Benj. Franklin Station, Washington, D. C.

[2] "Official Methods for Analysis of Malt." Published by the Malt Analysis Standardization Committee, 2374 Lincoln Avenue, Chicago, Ill.

The world fame of Danish beers such as Carlsberg, Tuborg, and others has largely been attributed to the fact that Danish brewers early recognized the value of scientific control in the malt and brewhouse. The laboratory was and still is one of the most important departments of the plant and due consideration is given to its decisions. Co-operative work of commercial interests with the Agricultural Station at Abed has resulted in the development of pure barley varieties of high brewing merit. Since 1923 pure barley strains are used exclusively, their main representatives being Binder and Opal; new types recently developed are Sterling, Isaria and Kenia. The Danish brewers request high quality brewing barley. Much attention is paid to the uniformity of kernel size and protein content which is not to exceed 10.5%.

Attempts to obtain a fair estimation of the brewing value of a barley from its chemical constituents have so far met with great difficulties. The determination of the starch content to estimate the extract of the malt appears to have some merit, however, the analytical methods are too complicated and time consuming to be of much practical value. The chemical analysis of the malt permits a more definite evaluation, although not a conclusive one.

In addition to the ordinary chemical analysis the Tuborg Breweries determine the enzymatic activity of cytase, protease, and diastase in the malt. The effect of cytase on the solution of the cell walls is estimated by the difference in extract between the fine and coarse grist of the malt. Proteolysis is measured by the percentage of salt-soluble proteins which is desired to be high. Further action of the proteases is determined by the amount of non-coagulable proteins in the wort—the higher the more thorough the proteolysis. The diastatic activity is measured by the time required for complete starch convertion as noted microscopically by the iodine test. The final effects of diastase are determined by sugar formation under standard conditions. The complete enzymatic action during mashing is expressed by examining the turbidity of the wort by means of a nephelometer giving the amount of colloids in the wort. Bright worts indicate proper enzymatic activity during mashing.

In the wet grains, that is the extracted malt after sparging, moisture, remaining extract, and non-converted starch are determined to check the mashing process.

The wort from the brewhouse is regularly examined for color, extract, and final attenuation, frequently for total protein content, proteins not precipitable with tannins, and formol protein. The final attenuation is used as a control method to ensure sufficient

fermentable substances in the beer when transferred into storage for effective after-fermentation.

The beer itself is examined for alcohol, carbon dioxide, and foam stability. For this latter property a simple method has been developed in the laboratory of the Carlsberg Brewery which gives the foam-forming and the foam-holding capacity in percentage of the total foam. The keeping quality, particularly important for export beers, is determined by placing the beer from 4 to 7 days into rather elaborate shaking devices duplicating the movement of ships and railroads. Turbidity and frothing of the beer are observed during this time and at the end of the period. These two beer defects are the most common and a great deal of research work centers around their elimination. Because of the interrelationship which exists between the colloids responsible for foam formation and those which may cause turbidity, this problem is a very difficult one. Good keeping qualities are partly based on the removal of those colloids which impart good foam to the beer. The cause for wild beer, that is beer shooting out of the bottle when opened, has not yet been defined, however, there is strong evidence to show that disturbances of the colloidal system are responsible. Rigid analytical control of all phases of the brewing process have yielded valuable assistance in this respect.

Furthermore, the Tuborg laboratory in particular has gone far beyond actual plant control. It renders valuable service in the economical utilization of waste products and idle machinery.

Research work in Danish breweries mainly centers around the fractionation of proteins, separating them into fractions precipitable with tannin and mercuric chloride, without, however, knowing the exact part of these fractions in beer production.

An entirely new feature which recently has been studied in brewing laboratories is viscosity. In Germany, Hoeffler has designed a special viscosimeter for brewing liquids. Some Danish investigators use this instrument while others use the ordinary Oswald viscosimeter. Hoeffler's work has indicated that the most famous beer types have unusually high viscosities. By testing the viscosimetric properties of the individual beer constituents he determined their contribution to the total viscosity of the finished beer. So far the dextrins have been found to have the highest viscosity. The nitrogenous substances have not been isolated for this purpose so far, but there are indications that their viscosity values will exceed those of the dextrins. These results are particularly valuable in the study of filtration problems.

The Carlsberg Laboratories, under the leadership of Prof. Soerensen, deal mostly with problems other than brewing problems.

Highly interesting is their work on micro-titrimetric methods for the determination of enzymes which permit 60 tests per day. One project now under way, of interest to brewing and malting chemists, is the study of the enzymatic activity during the growing of the malt sprouts. For most general enzymatic work the Willstaetter titration methods are used.

In the biological department of this institution, Dr. Nielsen has carried out an investigation which is interesting to the cereal chemist because of its relation to wort composition. In studying flocculation of the yeast during fermentation and storage, he found that it settles out more rapidly in worts containing large amounts of high molecular proteins. For this purpose he dialized the wort in two different portions. He proved that the yeast does not absorb these protein fractions by using a nutrient medium with a N content of 60–80%, the yeast showing not more than 3% of N in the dry substance.

Another development, although of more interest to the practical brewer, deserves to be mentioned here. There exists a strong tendency on part of the Danish brewers to decrease the alcoholic content of their beers in favor of improved aromatic properties. To this end research turns to the development of yeasts producing low alcoholic content but high in proteases which by thorough degradation of the proteins impart that much desired full-mouthedness in the beer.

In Germany the modification of malt is in the foreground of scientific investigation. Brewing chemists are particularly interested in establishing methods which will permit a numerical expression of this characteristic in place of the judgment of the practitioner as guided by bushelweight and physical properties. Fink, in Berlin, is investigating suitable methods for the determination of pentosans while Kolbach is giving his attention to the fractionation of proteins. His work on adsorption of certain protein fractions with activated carbon is well known from the literature. Fink has also developed a method for identifying yeast types in mixtures by means of the cytospectrum; based on this work are his attempts to transform one yeast type into another.

Both investigators, however, believe that other yet undefined substances, such as silica and the like, play an important part in the modification of malt.

In Munich, Dr. H. Leurs is conducting most interesting studies on the separation and identification of volatile fermentation products and on the nature of foam.

In France, Jean de Clerck has recently investigated the application of the oxidation-reduction potential to brewing control. His results

indicate promising trends, particularly for the study of those factors endangering the keeping qualities and favoring the growth of micro-organisms and the development of off-flavors in the finished beers, through the influence of light. The difficulty encountered in this study is that only a few of the involved oxidation-reduction systems are reversible and that the colorimetric methods do not give sufficiently accurate results, because the indicators may influence the tests. Electrometric methods are not practical because all samples have to be taken and all tests have to be conducted under the exclusion of air. Therefore, this feature is at the present only of academic interest, but it has some practical aspects for the future.

England has contributed much in recent years to the study of the correlation between the chemical analysis of barley and malt and their practical brewing value. Based on the pure barley strains available as the result of a ten-year developing program, Dr. Bishop has established definite relations between the various protein fractions and carbohydrate fractions of barley when the total protein and one fraction is known. He has also developed a formula which under certain definite conditions permits a fair estimation of the extract in malt from the protein content of the barley.

Starting from the knowledge that a correct adjustment of the pH of the wort affects the enzymatic activity favorably, Th. R. Dixon developed a new malt type which when used in amounts of 3% of the total malt increases the pH of the wort 0.1 to 0.2. This malt is known as enzymatic or proteolyt malt and has found favorable acceptance by the practical brewer. The principle of this new malt type is to increase the amount of proteolytic energy, obtained by treating the barley before germination with a pure culture of *B. Delbrucki*. The use of this malt is only a variation of the practice to correct the pH by lactic acid and has the advantage that its use requires less control. The processing method is comparatively simple and requires no special equipment. A more thorough proteolysis results in higher amounts of soluble and amino N which is of vital importance for yeast nutrition during fermentation. Other advantages claimed are better elimination of coagulable proteins, heavy cooler-deposits, higher extracts, and a more rapid clarification in the cellar.

Attenuation of the wort and flavor of the beer remain unaffected. Experiments were conducted on a large scale in Germany and an ethereal aroma was reported not only for the brew in which the enzymatic malt was used but also in following brews made with ordinary malts but pitched with the yeast from the first brew. This phenomenon was attributed to a probable esterification of the alcohols

during primary fermentation. The same principle is being applied in Germany where lactic acid cultures have been added to the malt, to the mash, and to the kettle wort with good results.

Fluorescence analysis is one of the latest developments of physical chemistry which is applied to cereal chemistry in general and its merits for the brewing laboratory in particular are studied by several English investigators.

REPORT OF THE 1934–35 COMMITTEE ON STANDARDIZATION OF LABORATORY BAKING

W. F. GEDDES, *Chairman*

Grain Research Laboratory, Board of Grain Commissioners, Winnipeg, Canada

(Read at the Annual Meeting, June 1935)

The Committee on the Standardization of Laboratory Baking appointed by the president for the past year consisted of T. R. Aitken, M. J. Blish, R. T. Bohn, D. A. Coleman, C. N. Frey, R. K. Larmour, G. Moen, E. B. Working, and W. F. Geddes.

Depressed finances and an increase in the volume of their regular laboratory work has rendered it particularly difficult for many of the members to carry out experimental studies in connection with the work of the Baking Test Committee. However, despite these unfavourable conditions, the spirit of co-operation and a deep interest in the Association has resulted in the presentation of eight Sub-Committee reports as listed below:

"Definition and Measurement of 'Flour Strength' as an Inherent Property of Wheat," M. J. Blish and R. M. Sandstedt.

"Study with the Regular Hobart-Swanson Mixer as Compared with the Hobart Mixer with Two Dough Hooks and the Cake Paddle for Mixing Bread Doughs," D. A. Coleman.

"Correlation of Experimental and Commercial Baking Tests," J. Freilich, S. McHugh and C. N. Frey.

"An Experimental Flour Mill for 100-Gram Wheat Samples," W. F. Geddes and B. Frisell.

"An Experimental Milling and Baking Technique Requiring 100 Grams of Wheat," W. F. Geddes and T. R. Aitken.

"Studies on Test Dough Mixer Calibration," Quick Landis and J. Freilich.

"The Effect on Gas Production of Substituting Various Salts for Sodium Chloride in the Baking Formula," R. K. Larmour and H. N. Bergsteinsson.

"A Further Study of the Determination of Flour Absorption by Means of the Supercentrifuge," E. B. Working.

In addition, the Baking Test Committee had the co-operation of the Research Committee of the Pioneer Section which undertook a

special study of baking pans and their report supplements the studies outlined above.

Recommendations

That a comprehensive study of baking tins, made of 2 XX tin, be undertaken on the influence of size and shape of pan and pan treatment, such as greasing and darkening, from the standpoint of the variability between replicates and differentiation between flours. Since the tall form tin is in general use, it should be employed as a reference standard.

That, in view of the fact that members are experiencing difficulty in securing the Hobart-Swanson Mixer, the Association should take this matter up with the Hobart Manufacturing Company.

That a further study be made of the method of reporting the results of the Standard Baking Test.

DEFINITION AND MEASUREMENT OF "FLOUR STRENGTH" AS AN INHERENT PROPERTY OF WHEAT [1, 2]

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(Read at the Annual Meeting, June 1935)

Platt (1934) aptly said that "nothing does advance a science more than clearcut ideas of what we are talking about." Cereal chemists have experienced considerable difficulty in attempting to reach a state of common understanding and agreement as to the manner in which flour "strength" should be defined. The term itself having no mutually acceptable definition or meaning, it is not surprising that standard and approved methods for identification, measurement, and interpretation have been found difficult to establish.

The prevailing tendency, apparently, is to accept the viewpoint expressed by Aitken and Geddes (1934), when they state that "protein content is recognized as being a reliable index to strength, when the wheats under consideration are of the same class," and that "when protein content is the only major variable, it is a reliable index of flour strength as reflected by loaf volume."

There is, however, a general unwillingness to accept protein content as a trustworthy index to strength when wheats of different classes or

[1] Subcommittee report, 1934–35 Committee on Standardization of Laboratory Baking.

[2] Published with the approval of the Director as Paper No. 167, Journal Series, Nebraska Agricultural Experiment Station.

varieties are under consideration, because variations in gluten quantity are alleged to be overshadowed by inherent differences in "quality."

That glutens do show inherent differences in their properties, as indicated by response to certain treatments and baking environments, cannot be denied. Furthermore, no one would question the need of reliable methods whereby these various properties can be distinguished, measured, and interpreted with reference to modern industrial practice.

In the endeavor to meet on common ground in defining and measuring inherent gluten properties, it is highly probable that one of the serious handicaps to progress has been the adoption and the persistent use of terms such as "strength" and "quality" in denoting these properties. These terms, themselves, imply comparative degrees of excellence or inferiority. The assumption that merely because two glutens have slightly different properties, one must of necessity be "better" or "stronger" than the other is decidedly unwarranted, and moreover it is not substantiated by actual experience in commercial practice. Nevertheless this premise has been made the basis for a large amount of investigational work, of which the main outcome has been only to verify the falsity of the assumption itself. This has been well illustrated, for instance, on occasions when several wheat varieties were submitted to different laboratories for appraisal and rating as to comparative "strength" or "quality." In studies of this type Swanson (1930), and Swanson and Kroeker (1932) found a pronounced disagreement amoung the different laboratories as to the order in which the varieties were rated. These divergent opinions obviously were merely reflections of different individual preferences, based to some degree upon the use of different methods of experimental baking.

Additional emphasis on the need for a generally acceptable or standard definition of "strength" is provided in recent studies reported by Aitken and Geddes (1934), and by Larmour and Brockington (1934). These studies were designed to test the idea that "strength" should be defined in terms of stability or tolerance toward various changes in baking environment, especially toward "severe" treatments. Using this criterion it was found that flours of lowest protein content had to be classed as the "strongest," because they showed the least loaf volume fluctuations in response to varying baking conditions. Such a conclusion is absurd, as pointed out by Aitken and Geddes (1934) themselves. Again, obviously, the fault is in the premise, not in the conclusion.

In the selection of a standard basis for defining flour strength it would seem rational to start from a point upon which there is concordance of opinion. All will agree that protein content is at least *to some degree* indicative of strength. The next step is, if possible, to establish

and define the conditions under which protein content, alone, is *not* a measure of strength. In this undertaking, the simplest practical path of approach is to consider that a flour of a given protein content should, in a suitable baking environment, produce a loaf of a certain volume within reasonable limits without any serious detriment to textural properties. Clearly, the higher the protein content, the greater the potentiality for loaf voume. When the test loaf fails to fulfill the expectations that its protein content would justify, it is likely to be suspected that the gluten was "weak," or of inferior quality. Experience has shown, however, that there is every justification for challenging the baking method rather than the flour itself. It therefore seems impossible adequately to define baking strength without reference to methods used for its ascertainment.

Applying these thoughts to the use of the standard A.A.C.C. baking test, and as has been emphasized, by Larmour and McCleod (1929), Geddes and Larmour (1933), and others, it is first of all necessary to recognize two distinct purposes for which flours are subjected to test baking. One involves the "control" testing of commercial flour with reference to its suitability for known requirements. The other is concerned with the testing of wheat varieties with reference to those *inherent properties* upon which judgement as to general commercial utility will ultimately be based. On the one hand we have flour presumably milled from established varieties, treated, processed, and ready for the bakeshop. On the other hand, we have experimentally milled flours, "green," unmatured, and conspicuously deficient in gassing power.

The important distinction is that in the case of the processed commercial flour the basic procedure is likely to furnish the necessary information, whereas with the experimentally milled flours, even after compensating for low gassing power, the basic procedure will in a large number of instances produce a small, undeveloped loaf having pronounced type J loaf tendencies, and this regardless of variety or protein content. Examination of a series of these test loaves, baked by the basic procedure, alone, would lead to the conclusion that protein content is in no sense a reliable index to baking strength. In this type of work, therefore, the supplementary tests are all important.

As applied particularly to experimentally milled, untreated flours, the bromate test is of outstanding value and importance, as has been so thoroughly established, confirmed, and verified, that references to specific studies are unnecessary. Indeed, Geddes and McCalla (1934) consider that in such work the bromate test should be accepted as the basic, rather than as a supplementary procedure. For the purposes of this discussion, the important feature of the bromate test is that when

it is applied to freshly milled untreated flours, under conditions of adequately maintained gas production, the effect upon gluten properties is such that response in terms of loaf volume is directly proportional to protein content. Other supplementary tests, involving bromate, that have been found similarly useful are the "blend-bromate" test (Larmour, 1931), the malt-bromate test, and the malt-bromate-phosphate test (Larmour, Geddes, and Whiteside, 1933).

Experience has shown that a flour which, in volume response to bromate treatment, will measure up to the capabilities indicated by its protein content, will also meet all reasonable *industrial* requirements in so far as "strength" or gluten "quality" is concerned. It is evident, therefore, that with such flours protein content is indeed a true measure of strength.

This situation clearly explains the value and the popularity of the bromate supplementary test as used for testing small lots of wheat, experimentally milled and untreated. In the opinion of the writers it has been far more indispensable for testing flour of this type than for testing commercially milled and processed flours, even though it was originally designed for use with flours of the commercial type. (See Werner and Herman, 1928.)

The tendency for experimentally milled flours to produce small, undeveloped loaves by the basic procedure, and to show a positive bromate response that is proportional to protein content is characteristic of certain well established southwestern hard winter wheat varieties, notably Kharkof, Kanred, and Blackhull. This raises the question as to whether wheats not conforming to this rule should be rated as "weak" or inferior.

At least two conspicuous exceptions are to be found in Tenmarq and Cheyenne. Tenmarq, a cross between Marquis and a sister strain of Kanred, characteristically gives a loaf of satisfactory volume and texture when baked by the basic procedure alone, but shows a negative response, or at the most a neutral response to treatment with bromate. Nevertheless, it is highly rated by the Kansas Experiment Station, and no doubt deservedly so, as a superior bread flour wheat. Apparently its lack of tolerance toward oxidizing agents is offset by two considerations. First, it needs no oxidation to produce a large, well-developed loaf. Secondly, it produces a flour of such superior color that only a minimum of artificial bleaching is required.

Werner and Herman (1928) consider that, in general, flours responding negatively to the bromate test "will be found lacking, under commercial usage, in what is termed by the practical baker strength or stability. As a rule they will not respond well to yeast foods or to severe treatment." Tenmarq is apparently an exception to this rule,

for in spite of its negative bromate response it has a very high tolerance to severe mixing (Swanson and Working, 1933). It may reasonably be concluded then, that although a positive bromate response is indicative of strength or of commercial utility, the reversed situation is not necessarily true.

Another instance of a wheat that would certainly be misjudged on the basis of the bromate differential test alone, is found in the comparatively new variety known as Cheyenne, a selection of Crimean Turkey, having superior agronomic features. This wheat is most unusual in its properties. When experimentally milled and subjected to the bromate differential test it gives a small type J loaf *both* with and without bromate, regardless of protein content, and regardless of the amount of bromate used. Attempts to produce the expected loaf volume by development in mixing, alone, did not succeed, although the flour showed exceptional tolerance and resistance to mixing.

As a result of the failure of Cheyenne wheat to show a loaf volume response proportional to its protein content either by the basic procedure, by the bromate test, or by mechanical treatment, it has on several occasions been rated as weak and of poor gluten quality. We have recently found, however, that when bromate and mechanical mixing development are *combined* in the same test, an excellent response is obtained. When a little shortening and malt are superimposed, the response is all that could be desired. The effects of these supplementary treatments, singly and combined, are clearly shown in the photographs and data which follow.

Figure 1 shows the small loaf volumes and lack of oven spring, not only by the basic, but by several *individual* supplementary procedures, even though the Cheyenne flour had a protein content of 13.5%. Figure 2 indicates the favorable response, in terms of volume, oven spring, and texture when suitable *combinations* of supplementary tests were used.

Figure 3 shows the potentialities of two Cheyenne flours of low protein content, the degree of response under suitable conditions being proportional to protein content. Certainly there is no indication of weakness or gluten inferiority when a favorable baking environment is provided.

These experimental results are typical of all Cheyenne samples thus far tested, and apparently are indicative of inherent and varietal properties not commonly found in the more familiar varieties of hard winter wheat. It is important to note that the combination of treatments necessary to bring out the full potentialities of Cheyenne is one that can be easily and readily provided in commercial practice.

Geddes and Larmour (1933) have said "It seems unlikely that we

shall ever find a single baking formula that can be relied upon to give an accurate estimate of flour value." Our own experience lends confirmation to this viewpoint, especially as applied to the varietal testing of small samples submitted by the agronomist and the plant breeder. In testing such samples for inherent "strength" or "gluten quality"

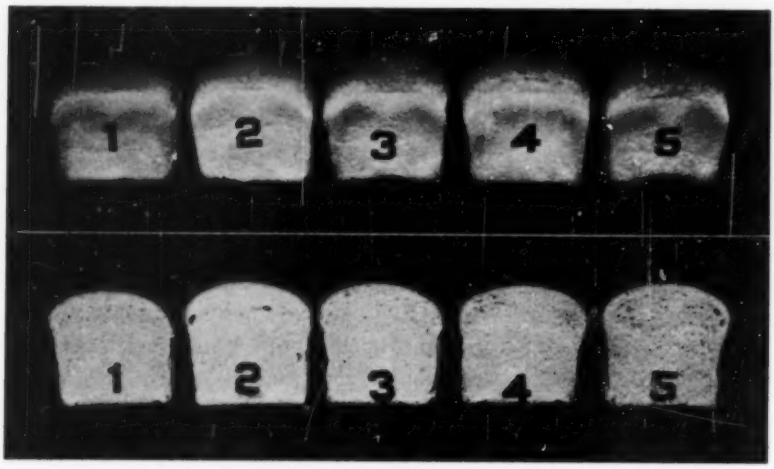

Fig. 1. Basic and individual supplementary tests with Cheyenne flour containing 13.5% protein.

1. Basic—loaf volume 490 cc.
2. Basic plus 1 mg. bromate—loaf volume 520 cc.
3. Basic plus 3 minutes mix—loaf volume 525 cc.
4. Basic plus 3% shortening—loaf volume 530 cc.
5. Basic plus 1% malt—loaf volume 530 cc.

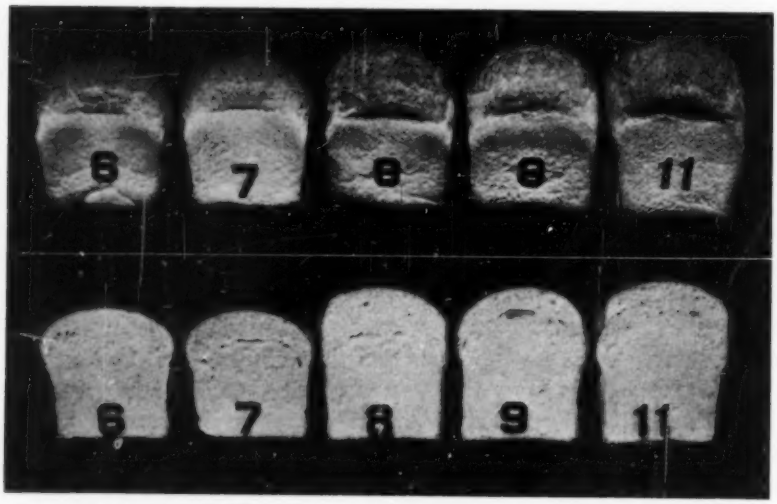

Fig. 2. Combinations of supplementary tests with Cheyenne flour containing 13.5% protein.

6. 3 minutes mix plus 3% shortening—loaf volume 600 cc.
7. 3% shortening plus 1 mg. of bromate—loaf volume 560 cc.
8. 3 minutes mix plus 1 mg. of bromate—loaf volume 660 cc.
9. 3 minutes mix plus bromate plus shortening—loaf volume 690 cc.
11. Mix plus bromate, shortening and, 1% malt—loaf volume 800 cc.

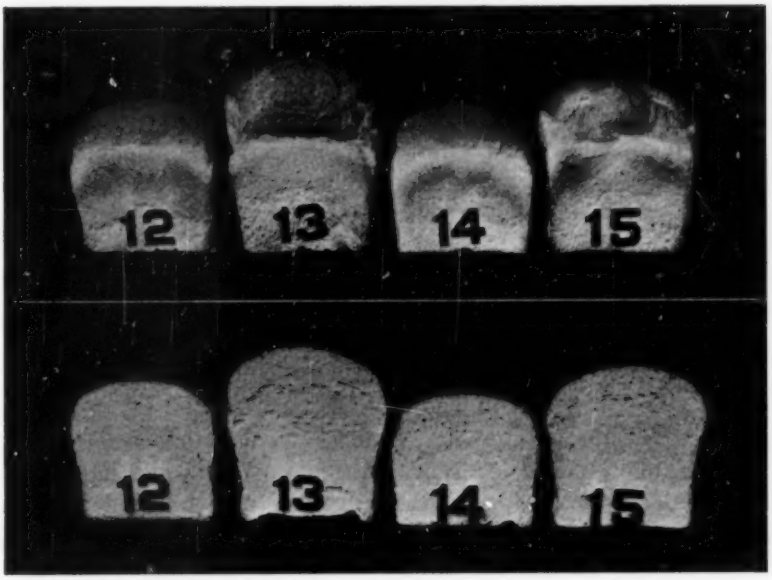

Fig. 3. Basic and combined supplementary tests with low protein Cheyenne flour.
12. 9.7% protein. Basic—loaf volume 450 cc.
13. 9.7% protein combined bromate, mix, shortening, malt—loaf volume 680 cc.
14. 9.0% protein. Basic—loaf volume 450 cc.
15. 9.0% protein. Combined bromate, mix, shortening, malt—loaf volume 615 cc.

the bromate test, or something equivalent to it, is the most valuable and informative of any *single* supplementary procedure, but with certain wheat varieties, such as Tenmarq and Cheyenne, a neutral or negative response to bromate, alone, does not signify weakness, inferiority, or lack of commercial utility. Both of these varieties, in response to treatments, either single or combined, that are easily available in commercial milling and baking practice, will readily exhibit potentialities proportional to protein content. Is this not, after all, the rational criterion of "flour strength"?

Applying this criterion to sound wheats of established varieties, including selections and hybrids of these varieties, experience leads more and more to the conclusion that, after all, *protein content and "strength" are one and the same thing, regardless of variety or of inherent differences in gluten properties.* In our own experience with this type of wheat testing, covering a period of several years, we have *in no single instance* found a sample or a variety that failed to measure up, in terms of volume and texture, to the potentialities that could be predicted from *protein content alone*, when provided with a suitable baking environment. Admittedly the optimum conditions were not the same for all varieties. Equally true, however, is the fact that no individual wheat or flour required any treatment or combination of

treatments not readily and *conveniently* available in modern industrial practice.

These laboratory experiences have been substantially verified in industrial experience, particularly with reference to southwestern hard winter wheats. All are familiar with the nature of complaints from the industry when new and unfamiliar varieties, such as Kanred, Blackhull, and even Turkey were introduced. In each case as increasing familiarity with certain special and inherent properties has been gained, the complaints have proportionately disappeared or diminished.

There is, of course, the possibility that the scope of our experience is too limited to permit the drawing of general and far-reaching conclusions. Nevertheless, it confirms the viewpoint previously stated by Werner (1925), when, as a result of extensive experience with commercial flours, he said "baking quality appears to be more a manifestation of chemical and physical environment in the process of baking than an inherent quality of the nitrogenous or other matter of wheat."

From these considerations, we submit that for all practical considerations any flour may be considered as "strong" as its *protein content* suggests. A flour is to be regarded as "weak" only to the extent that it fails to measure up in terms of loaf volume, textural properties, and tolerance, to the potentialities that would be predicted from its crude protein content in response to environments or treatments, single or combined, that are now *conveniently* available in modern commercial milling and baking practice. "Weak" flours are very rarely to be encountered, excepting in cases of unsoundness or of certain very special varieties known beyond any doubt to be unsuitable for commercial breadmaking.

We believe that not only is it entirely rational and logical to consider protein content and flour strength as inherently one and the same thing, but also that cereal technologists would find it to their advantage to go all the way, instead of only part of the way, in the acceptance of this viewpoint. The acknowledgment of this concept as being of general instead of limited application would tend greatly to simplify our approach to problems of common interest.

Mutual agreement upon this simplified definition of flour strength would mean, first of all, that inherent strength would be determined by the Kjeldahl method rather than by baking test. There would therefore be no more disputes and perplexities as to what particular baking test procedure provides the most reliable index to strength. The purpose of baking tests would be chiefly to determine the particular properties of the flour, and the treatment, lack of treatment, or combination of treatments necessary to render it suitable for modern

industrial requirements. The requirements may be general or specific, depending on the viewpoint of the technician.

Flour properties may be "inherent," or they may have previously been changed by artificial treatment. Therefore, when in the laboratory one undertakes to test an unfamiliar wheat variety for its *inherent* gluten properties, it is obviously desirable that the flour be freshly milled from sound wheat, and untreated with artificial maturing agents.

When such a flour is subjected to the standard A.A.C.C. baking procedure, the first necessary precaution is to insure the maintenance of adequate gas production, by proper adjustment of the sugar level, to the end that the gluten properties may have a chance to manifest themselves. Experimentally milled flours are almost certain to need 4 or 5 g., instead of 2.5 g. of sugar. Proceeding with the test from this point, it will be found that in the vast majority of cases the flour will fall into one of three classifications, the statement of these classifications being merely a summarization and reorganization of what has already been discussed.

The first and predominating class, in our experience, is the one in which the basic procedure produces a small, undeveloped type J loaf, regardless of protein content. With flours of this class the use of potassium bromate, or one of several other convenient oxidizers will bring about the desired "response" in terms of volume, oven spring, etc., the degree of response being proportional to protein content. When the protein content is extremely low, a neutral or negative response is to be anticipated. In the hard winter wheat area, as said before, the Turkeys, Kanreds, and Blackhulls typically fall in this class. For wheats of this class, the bromate supplementary test is accordingly of first importance. It is also highly desirable to know something of the flour's response and especially its *tolerance* toward mechanical treatment during dough mixing. This requires further supplementary tests in which the mixing time, alone, is systematically varied.

The second class may conveniently be designated as one in which the flour produces a loaf having good volume, oven-spring, and development when baked by the basic procedure alone. This flour obviously needs no special treatment, either chemical or mechanical, to bring out its potentialities. In view of the fact that such treatments, especially chemical, are habitually used in industrial practice, however, it is advisable to apply the same supplementary tests as were used for flours of the first class in order to test the flour's tolerance toward these treatments, and to indicate the degree to which they may be safely used in commercial milling and baking. Tenmarq is dis-

tinctly in this class. Marquis, in our experience, also frequently falls in this group when grown in Nebraska, the Dakotas, or Montana, although when grown under Canadian conditions it tends toward those properties characteristic of the first class.

The third class, which we have rarely encountered, and which does not seem to be generally recognized, is the one of which Cheyenne wheat is a typical representative. This, as we have shown, produces the small undeveloped type J loaf by the basic procedure. Regardless of protein content, it shows no satisfactory response, either to the bromate test, or to mechanical mixing, when each of these supplementary tests is used by itself, although it is extremely resistant to long mixing, as evidenced by failure to become sticky or "runny" even after several minutes in the Hobart-Swanson mixer. Response in proportion to protein content is however highly satisfactory when supplementary treatments are used, not singly, but in *combination*. Thus, as our accompanying data have shown, when oxidation with bromate is combined with an extended mixing treatment, there is a noteworthy response, and when shortening and a little malt are added to the combination the response is quite up to even the most optimistic expectations. Since the combination of treatments necessary to bring about optimum loaf characteristics is one readily and conveniently available in modern commercial practice, there is no justification for condemning such a flour as "weak," because it failed to "respond" either in the basic, or the bromate procedure, alone.

There is doubtless a fourth class, in which response is lacking, regardless of protein content, and irrespective of whether the flour is baked by the basic procedure, or by any supplementary treatments or combinations thereof that may conveniently be accorded in modern industrial practice. In so far as sound, undamaged wheats are concerned, we have never encountered one of this class among any of our established varieties, or among any selections or hybrids of these varieties. We are, therefore, disposed to adopt the simple viewpoint that, under American conditions, at least, inherent flour strength is synonymous with protein content, without the qualifications and reservations that are usually attached to this concept, and we propose this for the consideration of the A.A.C.C. Committee on Definitions.

The principles underlying the testing of untreated experimentally milled flours as to their inherent gluten properties, in terms of various responses and tolerances can also be safely applied to laboratory baking tests of all kinds, including mill and bake-shop control work. Here, however, both the procedure and the interpretation are ordinarily modified, and generally simplified, depending upon the purpose of the test, and upon the previous history and treatment of the flour. These

modifications and simplifications are so obvious that they need no detailed discussion.

In recommending the acceptance, without qualification or reservation, of the idea that protein content and inherent strength are one and the same thing, and that all flours of the same protein content have equal potentiality for producing a large, well-developed loaf (providing protein content is sufficiently high), there is no intended implication that all wheats of the same protein content should be regarded as of equal commercial value or utility. Even though color, milling properties, and flour yields may be the same, there can be, in any given instance, a warrantable basis for preference in favor of a certain variety or type, arising from habit, custom, and familiarity with the properties of that particular wheat. This is, however, merely a matter of convenience, and does not truly indicate that one wheat is inherently stronger or of better gluten quality than the other.

For the purpose under consideration, namely, the scientific testing of wheat and flour with reference to inherent "strength" or baking potentiality, every effort should be directed toward removing the influence of habit, custom, and preference from our definitions, and from our standard methods. The persistence of these factors has, no doubt, been the major source of confusion in our attempts to establish a rational and scientific, if not even a standard method of procedure for laboratory test baking.

Summary

Wheats of equal protein content have essentially the same baking potentialities, in terms of loaf volume, even though they do not all respond equally to any single baking formula and procedure. Therefore, for all practical purposes, protein content and inherent "flour strength" are one and the same thing. The only exceptions to this are to be found in wheats that fail to register responses proportional to their protein contents, when subjected to any treatments, *singly or combined*, that are nowadays at the convenient disposal of both miller and baker. Applying these criteria in our supplementary baking procedures, it will be found that such wheats are rarely, if ever, to be encountered among our established American wheat varieties intended for commercial bread production.

"Strength," as an inherent property, is to be determined not by the baking test, but by the protein test. The essential purpose of the baking test is to measure a flour's response to single and combined treatments and environments that are likely to be accorded to it under modern industrial conditions. All possible *combinations* should be tried, if necessary.

In terms of response to the basic and supplementary procedures provided by the standard A.A.C.C. experimental baking test, and used either singly or in combination, wheats may conveniently be classified into three, or at the most four, groups, with one of these groups predominating.

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STUDIES ON TEST DOUGH MIXER CALIBRATION[1]

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(Read at the Annual Meeting, June 1935)

Although the customary unit of mixing is the minute, it is recognized that each type of mixer, even each mixer of a specified type has its own characteristics. We have only indefinite indications that there is a difference in kind of development between the pull-tear type of treatment preponderant in nearly all multiple pin type mixers, and the pack-squeeze type which forms the principle action in a blade type mixer. All we can now definitely measure is the energy input, and since the amount of this required for maximum development is to a certain degree characteristic of a flour (mixed as a sponge), it offers for the present a reasonable basis for studies of calibration. It is well known that other factors influence the type or amount of mixing development received by a dough; consistency value, size, salt concentration, and temperature. It was found necessary to make a special water jacket for the pin type (Fleischmann) mixer used in these studies.

Methods

Two methods appear to be available for the determination of the end point, or the point at which what might be called maximum development is reached. When mixed as a sponge without salt in a multiple pin type mixer the dough gradually becomes smoother, but finally reaches a point at which it clings to the bowl, loses its tenacity and becomes of a very thick syrupy consistency. The Hobart mixer with one dough hook or paddle appears to produce a non-uniform development, the lower part of the dough breaking down before the upper part has had appreciable development. Again when tested in the Farinograph (as a sponge) the consistency rises to a maximum and then drops, the width of the curve also decreasing. This appears to correspond to the breakdown of the dough in the conventional mixer. We have used the Farinograph as a standardizing instrument, since with it reproducible values of consistency can be readily obtained.

[1] Subcommittee report, 1934–35 Committee on the Standardization of Laboratory Baking.

Farinograph standard

Obtain the absorption (at 500° B.) and test curve of any typical patent bread flour, preferably one with fairly low total Development Energy (D.E., see Landis, 1935).[2] Weigh out that amount of flour for which the calibration is to be obtained. Mix in the mixer to be calibrated, several samples for varying periods of time. In case the amount of flour is less than 300 g. it is necessary to mix two or more successive batches as nearly alike as possible, then combine and scale off an aliquot equivalent to 300 g. flour. This is immediately tested in the Farinograph, mixing until the peak in the curve has been reached and passed. Determine the remaining D.E. by taking the area under the curve. Subtract this from the total energy previously determined. The result must be assumed to be the D.E. imparted by the mixer under calibration for the corresponding time interval. In a typical instance the results in Table I were obtained.

TABLE I

TYPICAL RESULTS ON CALIBRATION WITH FARINOGRAPH STANDARD[1]

Mixing time min.	Remaining D.E. kilo units	D.E. from test mixer kilo units	D.E. rate kilo units/min.
2	2.46	1.44	0.72
3	1.88	2.02	0.67
5	0.95	2.95	0.59
6	0.51	3.39	0.57
		Average	0.66

[1] Farinograph titration of flour; 191 cc. at 3 min., absorption 63.7%. Total D.E. 3.90 kilo units.

The theoretical rate of input of D.E. for the Farinograph at 500° B. consistency is, of course, 0.50. Actually it is found to be slightly less; about 0.46 D.E. per minute. Hence it may be concluded that the test dough mixer for which these figures are given will develop the dough at an average rate of 0.66/0.46 or 1.43 times as fast as the Farinograph. A summary of the results obtained to date by this method are given in Table II.

TABLE II

SOME AVERAGE VALUES FOR MIXER CALIBRATION FOR 300 G. FLOUR

Mixer	Speed	Calibration value, kilo units per min.
Farinograph	60 r. p. m.[1]	0.46
Fleischmann	105 r. p. m.	0.66
Hobart:		
one dough hook	No. 1	0.14
" " "	No. 2	0.17
" " "	No. 3	0.39
paddle	No. 2	0.19
"	No. 3	0.53

[1] Slow blade. The speed of the other blade is 90 r. p. m.

[2] Landis, Quick. Index of proteolytic activity by the use of the Farinograph. Cereal Chem. 12: 25–33 (1935).

There seems to be a tendency for the average rate of D.E. input to decrease as mixing proceeds. We believe that this is partly due to unavoidable temperature rise in the dough during mixing as well as to the fact that the average values include the time required for preliminary incorporation of the ingredients. Actually, at constant temperature, the consistency gradually rises until the peak of development. Hence it is advisable to use a rapidly developing flour with relatively low D.E. value in cases where mixing temperature cannot be controlled, allowing the peak to be approached before excessive temperature rise occurs.

Comparison standard

If the calibration for one mixer is known, or the D.E. value for a standardized flour is determined, it should be possible to calibrate another mixer from them (except perhaps mixers like the Hobart with one dough hook or paddle; *vide supra*). In this method no absolute reference standard of consistency can be immediately obtained, hence the absorption should be carefully controlled and the flour, if standardized, kept in a sealed container. The absorption value should accompany the sample. For patent flours with low ash and no added salt the breaking point of the dough can readily be determined within ½ minute. If the D.E. of the flour is known, a simple calculation gives the average value of the mixer calibration in D.E. per minute. If it is not known, it can be tentatively assumed (see values below which support this assumption) that these values are in inverse ratio to the times to breakdown, and a comparison with a mixer whose calibration is known can be made. For example, the time to breakdown in the Fleischmann mixer used for the above data for a particular flour was 10 minutes. In the Farinograph (visual observation of the dough) it was 13 minutes. Hence the ratio is 13/10 or 1.3, and since the Farinograph constant is 0.46 as actually determined, the value for the mixer under test is $1.3 \times 0.46 = 0.60$. This is slightly lower than the average value of 0.66 determined by Method I, but may be explained by the unavoidable temperature rise which occurs when the dough is mixed to destruction. Although a water jacket has been installed around the mixing bowl, a temperature rise of nearly 5° F. still occurred in the 10 minutes of mixing, but without the jacket the rise was over 12° F., completely invalidating the results.

Summary

Two methods of calibration are described; (1) use of the Farinograph as a direct calibration instrument, (2) a mixer of known calibration or a previously calibrated flour of known D.E. value as indirect comparison.

CORRELATION OF EXPERIMENTAL AND COMMERCIAL BAKING TESTS [1, 2]

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(Read at the Annual Meeting, June 1935)

Introduction

The problem of the correlation between laboratory baking tests and commercial results is one of fundamental importance to those engaged in laboratory as well as large scale baking. A vast amount of laboratory control and research work has been done in the past, is being done at the present time, and will be done for a long time to come, unless more valuable or simpler chemical and physical methods are developed to replace the baking test. The results of such laboratory baking tests have proven themselves of great value, and leaders in the milling, baking, and allied industries appreciate this, as evidenced by their continued support of laboratory work, and their interest in the work of the A.A.C.C. Baking Test Committee.

It is therefore not necessary, as some critics or sceptics contend, to justify the existence and use of the baking test. On the other hand, it cannot be denied that the results of baking tests have at times appeared to be of questionable significance.

Among the reasons for this may be mentioned the mechanization of the baking procedure, and changes in the type of formula and method used in large scale bread production, in which particulars laboratories have not always kept pace with developments in the bakeries. This has increasingly emphasized the importance of a proper interpretation of laboratory results, and involves the danger of taking too many liberties in making such interpretations. It would be preferable to bring laboratory apparatus up to date, and to modify formulae and methods with a view to minimizing the necessity for a broad interpretation.

It is, of course, not our intention to underestimate the necessity for a sufficient knowledge of the fundamental principles and factors involved in the processes of bread-making, which knowledge would

[1] Subcommittee report, 1934–35 Committee on the Standardization of Laboratory Baking.

[2] We wish to acknowledge the valuable assistance given us by Mr. G. H. Ekstedt of our Bakery Unit who conducted the commercial baking operations.

make the interpretation of laboratory baking results much more accurate, regardless of the type of procedure or apparatus used in making such baking tests.

The work described below was undertaken in order to see what correlation actually exists between laboratory and large scale baking results, and it is hoped, will indicate how to make the results of laboratory baking tests more valuable than they have been in the past.

Nature of the Problem

The problem may be broadly divided into two parts:

First—Correlation between methods of procedure.

Second—Correlation between the kind and degree of differences obtained when making comparative tests between different doughs in the laboratory, compared to the differences obtained when making similar tests in the bakery.

These parts may again be subdivided; the first part, for instance, into sponge and straight doughs, and each of these into the different steps involved—such as mixing, fermentation, etc. The individual steps, such as mixing, may in turn be subdivided into the factors involved—such as type of mixer, type of formula, size of dough, time, temperature, etc.

It is evident that the problem is a very broad and all inclusive one. With only a limited time at our disposal, we had to confine ourselves to a very small portion of the work involved in the above outline.

It was decided to limit the work to straight doughs, and particularly to this question: Is it possible, under laboratory conditions, to produce a loaf of bread so like a commercial loaf in character, that it would be impossible to distinguish between them when placed side by side and judged by the usual methods? In other words, is it possible to mix, ferment, bake, etc., the same kind of loaf in the laboratory as in the bakery? If not, what step or steps in the baking procedure would make it possible?

We, therefore, undertook a study of the different steps in the straight dough procedure with this question in mind, and starting with mixing, went through every step in the baking procedure, varying only one factor in each experiment.

Formula and Procedure

It was decided to use an average straight dough formula, typical of current commercial practice, and the dough was put through the usual bakery procedure. The formula and method used in the bakery were as follows:

Flour	100%
Water	65%
Sugar	6%
Salt	2%
Lard	3%
Yeast	2%
Malt extract	1%
Skim milk powder	4%
Yeast food (Arkady)	½%

The flour used was a blend of three widely used commercial patents.

The dough was mixed for 10 minutes, came out of the mixer at 80° F., and was fermented for 2¼ to 2½ hours in a room maintained at 80° F.; the dough was not punched. After fermentation, the dough was put through the divider and rounder, given 12 to 15 minutes bench proof, put through the molder, proofed in the pans at 95° F., and baked for 35 minutes in an electrically-heated oven in which the initial temperature was 500° F., and the temperature during most of the baking period ranged around 450° F. Steam under 15 lbs. pressure was admitted into the oven during the first few minutes of baking.

The following diagram indicates the method of study used.

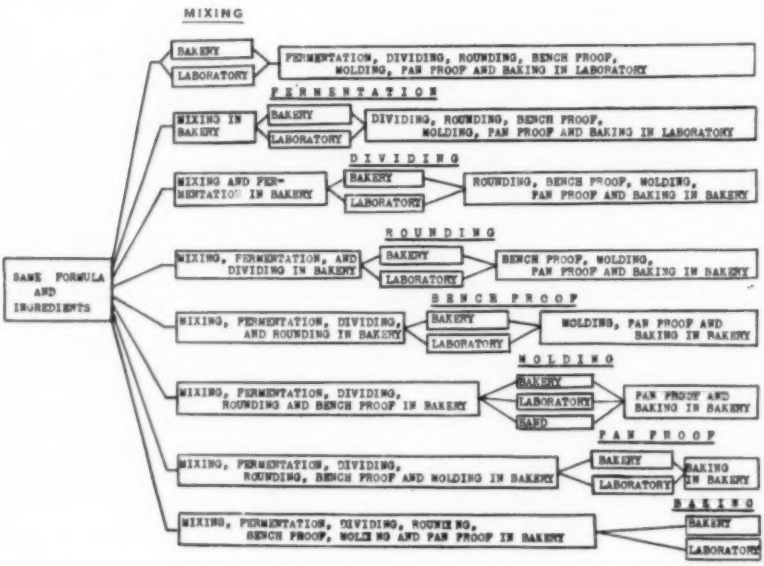

Fig. 1. Diagram indicating method used in comparing bakery and laboratory results for the individual steps in the baking procedure.

Mixing

In conducting the mixing experiments, the Brabender Farinograph was used as a guide. Doughs from the bakery and laboratory mixers were placed in the Farinograph for a few minutes and the resulting curves compared.

The following curves were obtained for the bakery dough, and for laboratory doughs mixed for varying lengths of time in the laboratory mixer.

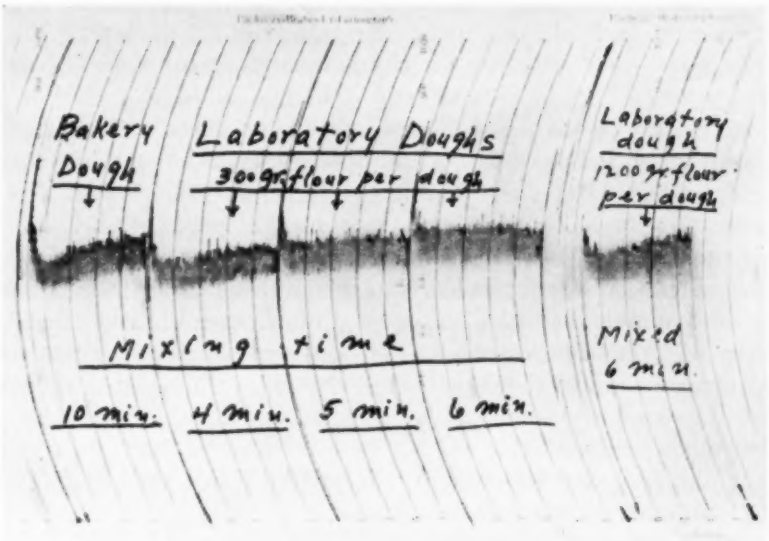

Fig. 2. Farinograph curves produced by doughs mixed in bakery and in laboratory mixers.

These curves indicated that 4 to 5 minutes mixing in the laboratory mixer would be equivalent to 10 minutes mixing in the large bakery mixer.

With these figures in mind, doughs mixed for varying lengths of time in the laboratory mixer were made into bread and compared to bread made from bakery mixed dough. After mixing, both bakery and laboratory doughs were fermented, molded, and otherwise handled in exactly the same way in the laboratory, so that mixing was the only variable. Molding was done in a Thomson machine.

In the following tests, mixing times in the laboratory were varied from 4 to 12 minutes, in two-minute increments:

	Mixing time	Dough time	Pan proof	Volume per pound of bread
	Min.	*Min.*	*Min.*	*Cc.*
Bakery dough	10	142	44	2360 [1]
Laboratory dough	4	137	47	2320
	6	141	47	2420
	8	140	46	2420
	10	150	46	2400
	12	147	47	2410

[1] Average of three loaves.

In crust color and exterior characteristics, these loaves were all about alike.

In crumb color, the 4-minute mixing produced a loaf slightly darker than the bakery bread, 6-minute mixing was equal to the bakery bread, and, 8-, 10-, and 12-minute mixing produced loaves slightly whiter than the bakery bread.

In texture, the 4-minute loaf was not quite as fine grained as the bakery bread, the 6-minute loaf was equal to the bakery bread, the 8-minute loaf was slightly more open grained, and 10- and 12-minute loaves still more open grained. Thus we see that both crumb color and texture characteristics indicated that about 6 minutes mixing in the laboratory mixer came nearer to equaling bakery mixing than any other mixing period.

In the following experiment, the mixing times were varied from 3 to 7 minutes, in one-minute increments:

	Mixing time	Dough time	Pan proof	Volume per pound of bread
	Min.	*Min.*	*Min.*	*Cc.*
Bakery dough	10	140	39	2250 [1]
Laboratory dough	3	138	40	2180
	4	139	43	2260
	5	137	40	2230
	6	138	38	2320
	7	144	42	2320

[1] Average of two loaves.

In crust color and exterior appearance the loaves were again about alike. In crumb color, 3 minutes mixing made the loaf darker, 4 minutes slightly darker, and 5 minutes equal to the bakery bread, while the 6- and 7-minute loaves were slightly whiter. In texture, the 3-minute loaf was not as fine grained as the bakery bread, the

4-, 5-, and 6-minute loaves were about equal to the bakery bread, and the 7-minute loaf slightly more open grained.

These experiments indicated that 5 or 6 minutes in the laboratory mixer would produce bread very similar in character to bread made from bakery mixed dough, if all other factors were kept constant.

Fermentation

In the fermentation experiments, only bakery mixed dough was used. After the large dough came out of the bakery mixer, it was placed in the trough and fermented in the usual way, while a few pounds of it were taken to the laboratory and divided into portions of 550 g.—which is equal to the weight of dough mixed per loaf for this formula. These small doughs were fermented for varying lengths of time under laboratory conditions—in glass battery jars, partly immersed in water in a cabinet maintained at 86° F. and constant humidity (compared to 80° F. for the large bakery dough). This temperature (86° F.) was used in preference to 80° F. because laboratory fermentations were usually carried out at that temperature, and a very carefully controlled water bath was available for the purpose.

After fermentation, these doughs, and a few portions of bakery fermented dough, were handled and baked in exactly the same way in the laboratory.

The fermentation times and loaf volumes for one experiment were as follows:

	Dough time	Pan proof	Volume per pound of bread
	Min.	*Min.*	*Cc.*
Dough fermentation in bakery at 80° F.	155	45	2310[1]
Dough fermentation in laboratory at 86° F.	90	55	2300
	120	48	2400
	150	40	2380
	180	34	2310

[1] Average of two loaves.

The 90-minute fermentation produced a loaf with a slightly rough break. The other loaves all had smooth breaks. There was not much difference in crumb color between any of the loaves. In texture, the 90-minute loaf was slightly closer grained than the bakery bread, the 120- and 150-minute loaves were very similar to the bakery bread, and the 180-minute loaf was much more open grained than the bakery bread.

These results indicated that 120 to 150 minutes fermentation under laboratory conditions would approximately equal 155 minutes of bakery fermentation.

The following are the fermentation times and loaf volumes for another fermentation experiment:

	Dough time	Pan proof	Volume per pound of bread
	Min.	*Min.*	*Cc.*
Dough fermentation in bakery at 80° F.	155	51	2290 [1]
Dough fermentation in laboratory at 86° F.	105	58	2260
	120	56	2330
	135	54	2320
	150	49	2370
	165	49	—

[1] Average of three loaves.

There was not much difference between any of these loaves in exterior characteristics.

In texture, the 105-minute loaf had slightly thicker cell walls than the bakery bread, while the other laboratory fermented loaves were similar in character to the bakery bread, with the 150-minute loaf slightly better than the others. (See Figure 3.)

These experiments indicated that when the doughs were handled in the laboratory, other things being equal, a bakery fermentation of 155 minutes at 80° F. could be approximately equaled by laboratory fermentation of from 120 to about 150 minutes at 86° F.

In the dividing, rounding, bench proof, molding, pan proof, and baking experiments, only bakery mixed and fermented dough was used.

Dividing

After fermentation, a portion of the dough was taken just before it went through the divider, and scaled by hand into pieces equal in weight to those which were being scaled by the divider from the main body of the dough. After scaling and dividing, all the pieces went through the usual bakery procedure in the same way.

The volume figures for two such experiments were as follows:

	Volume per pound of bread (Average of three loaves)	
	Expt. No. 1	Expt. No. 2
	Cc.	*Cc.*
Divided in bakery, by divider	2260	2120
Scaled by hand, Laboratory method	2340	2180

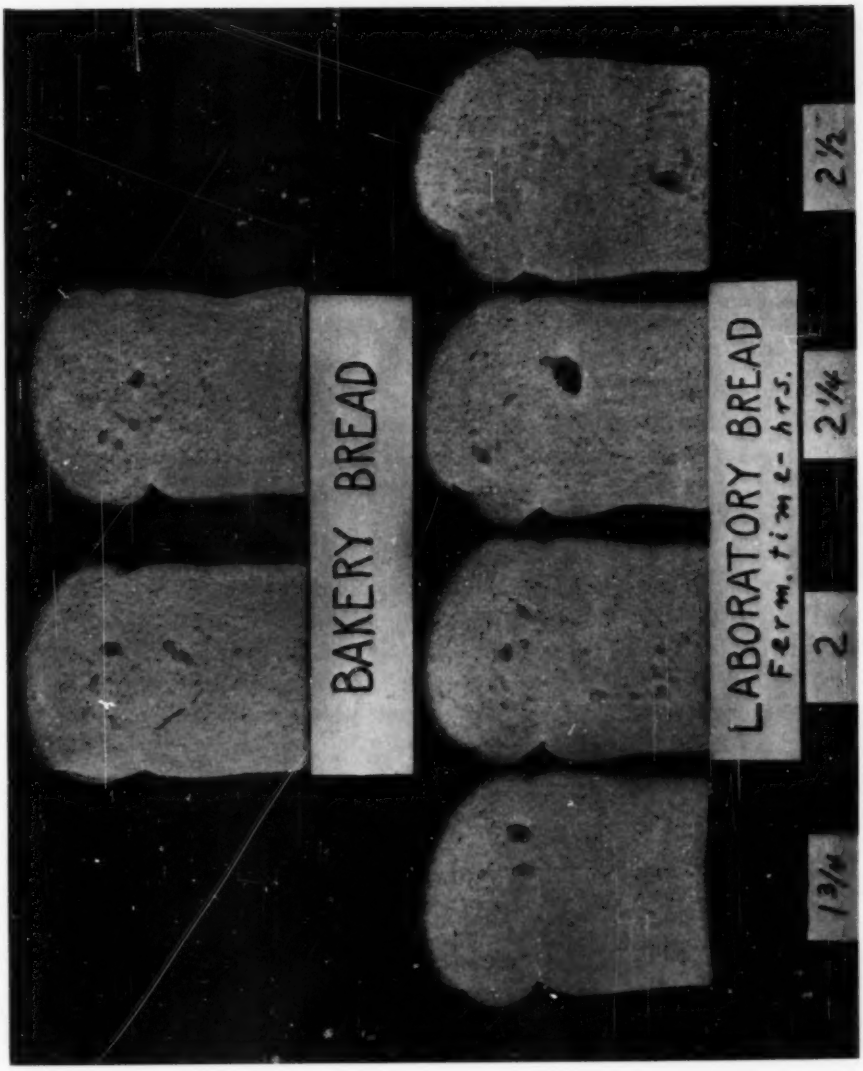

Fig. 3. Photograph showing a series of loaves fermented for different lengths of time in laboratory compared to loaves fermented 155 minutes in bakery.

In both experiments, the external and internal characteristics of the bakery divided loaves were quite similar to those which were scaled by hand.

Rounding

After the dough pieces came through the divider, some were rounded by hand as the rest were going through the rounding machine, after which all were handled in exactly the same way. The following volumes were obtained in two such experiments:

	Volume per pound of bread (Average of three loaves)	
	Expt. No. 1	Expt. No. 2
	Cc.	*Cc.*
Rounded in bakery	2260	2120
Rounded by hand, Laboratory method	2220	2140

The volume differences are small and inconsistent, and may therefore be considered within experimental error. The external and internal characteristics were similar in both cases, and any small differences observed were apparently within experimental error.

Bench Proof (Intermediate Proof)

After the doughs came through the rounder, some of the pieces were taken to the laboratory and set in the 86° F. cabinet, while the others were kept in boxes in the bakery for the duration of the bench-proof period, after which all were handled the same way in the bakery.

The volume figures for this experiment were as follows:

	Volume per pound of bread (Average of three loaves)
	Cc.
Bench proof in bakery	2260
Bench proof in laboratory	2320

These loaves were very similar in external and internal characteristics, but the laboratory loaves were slightly more open grained—evidently due to somewhat greater volume.

Molding

After the bench-proof period in the bakery, some of the dough pieces were molded in the bakery molder—the new type Thomson molding machine, some were molded in the laboratory molder—the old type Thomson machine, and some were molded by hand. After

molding, all doughs were proofed and baked in the same way in the bakery.

The volume figures for two molding experiments were as follows:

	Volume per pound of bread (Average of three olaves)	
	Expt. No. 1	Expt. No. 2
	Cc.	*Cc.*
Bakery molder used	2120	2240
Laboratory molder used	2230	2110
Molded by hand	2180	2200

Oddly enough, hand molding gave the most consistent loaf volumes. The variations in machine molding were probably due to the fact that the roller and drum plate adjustments on both molders were changed for each experiment.

In the first experiment, the bakery molder settings were too narrow, and this, coupled with the fact that the dough was slightly soft and slightly over-aged, produced bread of rather poor texture, compared to that molded by the laboratory molder, in which the settings were wider, so that the dough received less punishment.

In the second experiment, the distances between the rollers were made exactly alike on both molders, and the drum plate distances were nearer alike than in the first experiment. Although still different in volume, the resulting loaves were very similar to each other in internal characteristics, and much better than those which went through the bakery molder in the first experiment. This emphasized the importance of proper and uniform molder adjustments in experimental work.

The hand-molded loaves were just about alike in character for both experiments, but quite different from the bread molded by either of the machines, in both internal and external characteristics. It did not look like machine-made commercial bread.

Pan Proof

After coming through the bakery molder, some of the doughs were proofed in the laboratory cabinet at 100° F., while the rest were being proofed in the bakery at 95° F. After proofing all the doughs went into the bakery oven at the same time, and were baked for the same length of time.

In two pan-proof experiments, the volume figures were as follows:

	Volume per pound of bread	
	Expt. No. 1 (Average of 3 loaves)	Expt. No. 2 (Average of 6 loaves)
	Cc.	*Cc.*
Pan proof in bakery	2120	2310
Pan proof in laboratory	2120	2280

The volume differences between bakery and laboratory proofing are evidently insignificant. The external and internal characteristics were also very similar when comparing the bakery and laboratory proofed bread.

Baking-Oven Tests

There are two ovens in the laboratory, one of which is equipped with a rotating tray arrangement for more uniform heating conditions; but the trays in this oven were too small to hold the large bakery pans. It was therefore decided to use the other oven, so that the bread baked in the larger bakery pans in this oven could be compared with the bread baked in the same sized pans in the bakery oven.

All oven tests were made with bakery doughs; those baked in the laboratory oven were proofed either in the bakery or laboratory, as indicated in the table. The laboratory oven used was a Freas Electric Oven, with bottom heat only, and equipped with two fans for circulating the air in the oven—one fan near the bottom on one side of the oven, and another fan near the top on the other side of the oven. This oven also had some ventilating holes on top which were open or closed as indicated in the table. The temperature in the laboratory oven was 428° F. and in the bakery oven 500° to 450° F. Steam was, or was not, used in the bakery oven as indicated in the table. Shallow pans of water were placed against the back and sides within the laboratory oven. Conditions were altered in the ovens in an attempt to find out under what conditions laboratory baked bread would most closely resemble bread baked in the bakery oven.

Table I shows the conditions used and the loaf volumes obtained in a number of experiments.

The results of these baking experiments were as follows:

In crust color, the laboratory baked loaves were generally darker on top, and lighter on the sides and bottom than the bakery loaves. No two laboratory loaves were alike, and no two sides of any one loaf were alike in color. When no steam was used in the bakery oven, the bakery bread was somewhat similar to laboratory baked bread in

crust color. The laboratory loaves were generally less uniform in color on top and more uniform on the bottom and sides, compared to the bakery loaves.

TABLE I

RESULTS OF BAKERY AND LABORATORY OVEN EXFERIMENTS

Experiment number	Baked in	Pan proofed in	Oven conditions[1]	Volume per pound of bread (Av. of six loaves)
				Cc.
1	Bakery Laboratory	Bakery Bakery	B. No steam L. Holes open, both fans used	2310 2210
2	Bakery Laboratory	Bakery Laboratory	B. No steam L. Holes closed, lower fan only used	2390 2340
3	Bakery Laboratory	Bakery Laboratory	B. Steam used L. Holes closed, top fan only used	2270 2340
4	Bakery Laboratory	Bakery Laboratory	B. Steam used L. Holes closed, both fans used	2280 2280
5	Bakery Laboratory	Bakery Laboratory	B. Steam used L. Holes open, both fans used	2230 2310

[1] B. = Bakery.
L. = Laboratory.

In exterior appearance the laboratory loaves were either similar to or had slightly smoother breaks than the bakery bread.

The laboratory bread was generally darker in crumb color and more open grained in texture than the bakery bread.

In no case was the laboratory baked bread similar enough to the bakery baked bread, in internal or external characteristics, to make it difficult to distinguish between them, although there was some similarity in a general way.

Mixing and Fermentation Combined

Having thus far studied each step in the baking procedure alone, a number of experiments were now made combining mixing and fermentation,—steps in the procedure which, according to the experiments described above, could be adjusted to give the same results in the bakery and the laboratory. The following diagram indicates the method used.

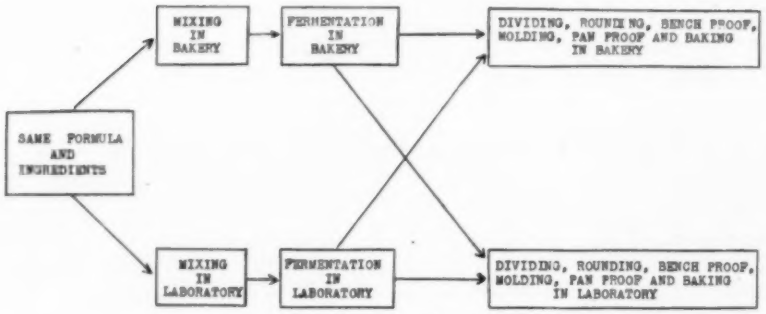
MIXING IN BAKERY
FERMENTATION IN BAKERY
DIVIDING, ROUNDING, BENCH PROOF, MOLDING, PAN PROOF AND BAKING IN BAKERY
SAME FORMULA AND INGREDIENTS
MIXING IN LABORATORY
FERMENTATION IN LABORATORY
DIVIDING, ROUNDING, BENCH PROOF, MOLDING, PAN PROOF AND BAKING IN LABORATORY

Fig. 4. Diagram indicating method used when comparing results of combined mixing and fermentation in laboratory and bakery.

In order to have a large enough laboratory dough to put through the bakery divider, after mixing and fermentation in the laboratory, it was necessary to mix and combine two four-loaf batches—using 1200 g. of flour to each batch (300 g. flour per loaf is used in regular laboratory work). This produced almost 10 lbs. of dough, which gave 6 to 7 dough pieces after going through the divider.

After mixing and fermentation in the laboratory, this 10-lb. dough was taken to the bakery and put through the divider immediately after the bakery dough; from this point on, both the bakery and laboratory doughs were handled in exactly the same way. It was therefore necessary to properly time laboratory operations in order that the laboratory dough might be ready immediately after the bakery dough. This was more difficult than anticipated, and in the first experiments the laboratory dough was either too old or too young, as shown below.

The mixing, fermentation time, and volume figures in the first of these experiments were as follows:

Dough mixed and fermented in	Mixing time	Dough time	Bench proof	Pan proof	Volume per pound of bread (Average of 7 loaves)
	Min.	*Min.*	*Min.*	*Min.*	*Cc.*
Bakery, at 80° F.	10	140	13	66	2390
Laboratory, at 86° F.	6	135	13	62	2380

The loaves made from the dough which had been mixed and fermented in the laboratory were darker in crumb color, as a group, than the bakery loaves. They were also more open grained, and seemed to have older dough characteristics, compared to the bakery

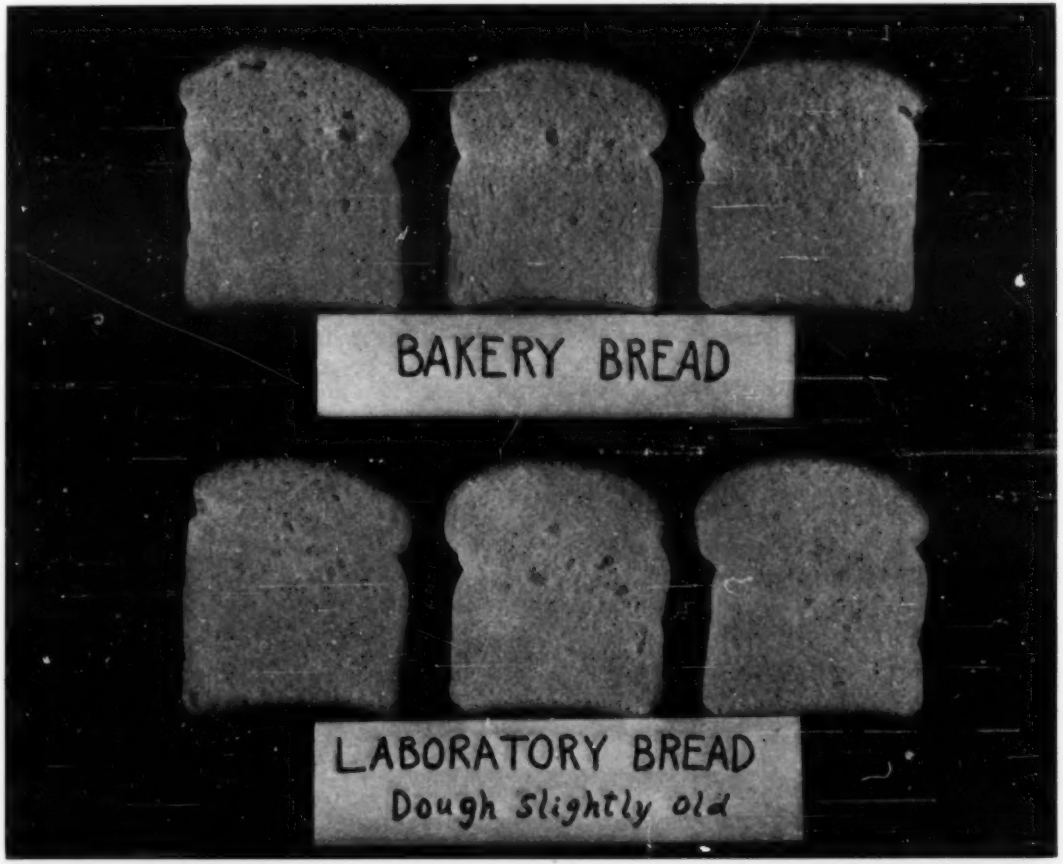

Fig. 5. Bread made from doughs which were handled in the bakery in exactly the same way, after combined mixing and fermentation in the bakery, compared to combined mixing and fermentation in the laboratory. Laboratory dough too old.

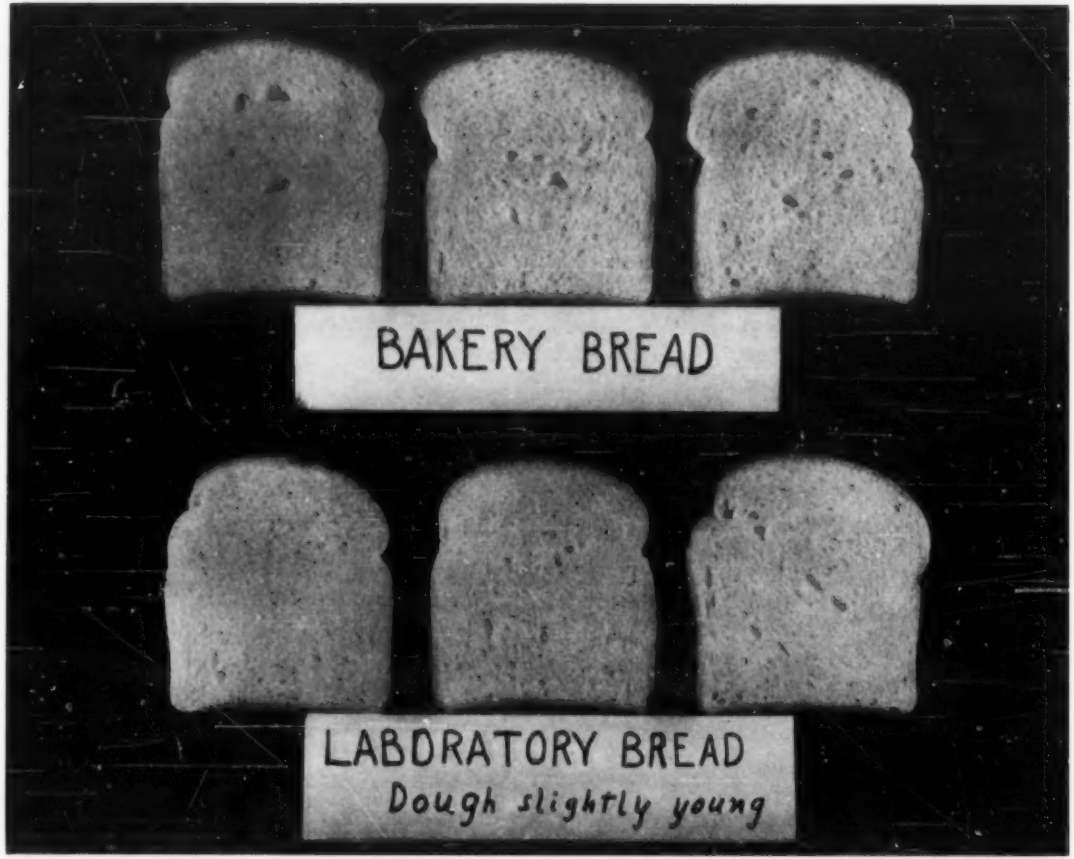

Fig. 6. Bread made from doughs which were handled in the bakery in exactly the same way, after combined mixing and fermentation the bakery, compared to combined mixing and fermentation in the laboratory. Laboratory dough too young.

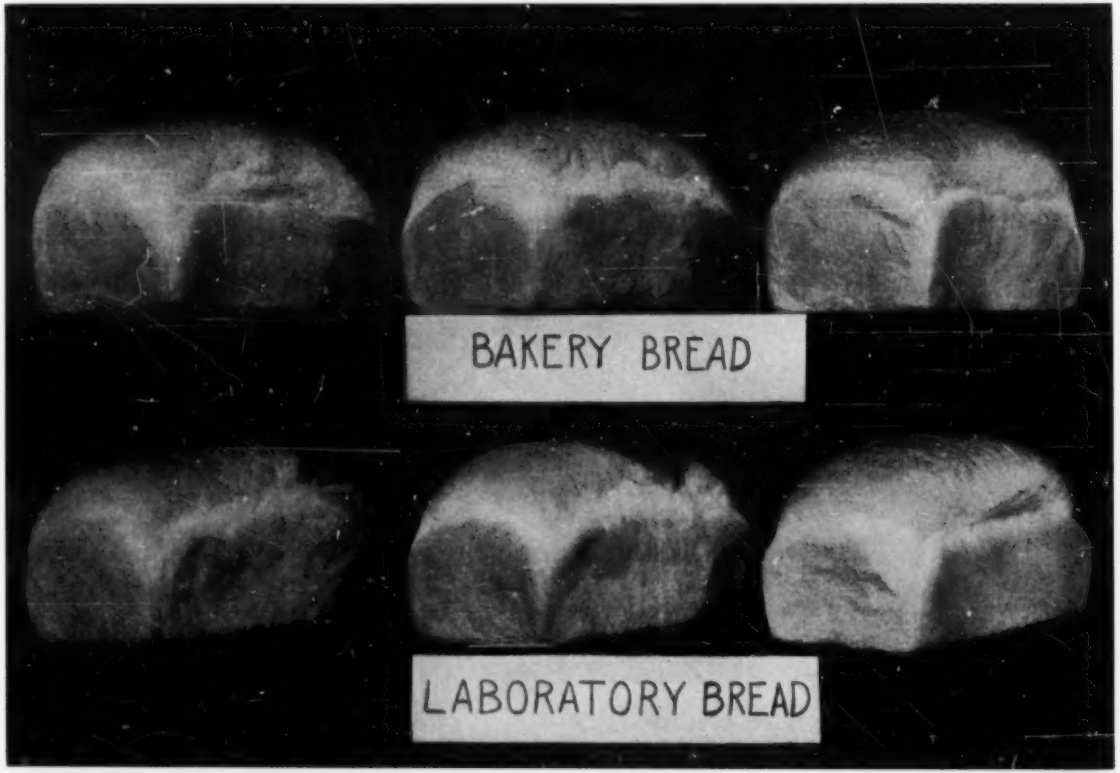

Fig. 7. Bread made from doughs which were handled in the bakery in exactly the same way after combined mixing and fermentation in the bakery, compared to combined mixing and fermentation in the laboratory. Exterior views; bakery and laboratory loaves practically alike.

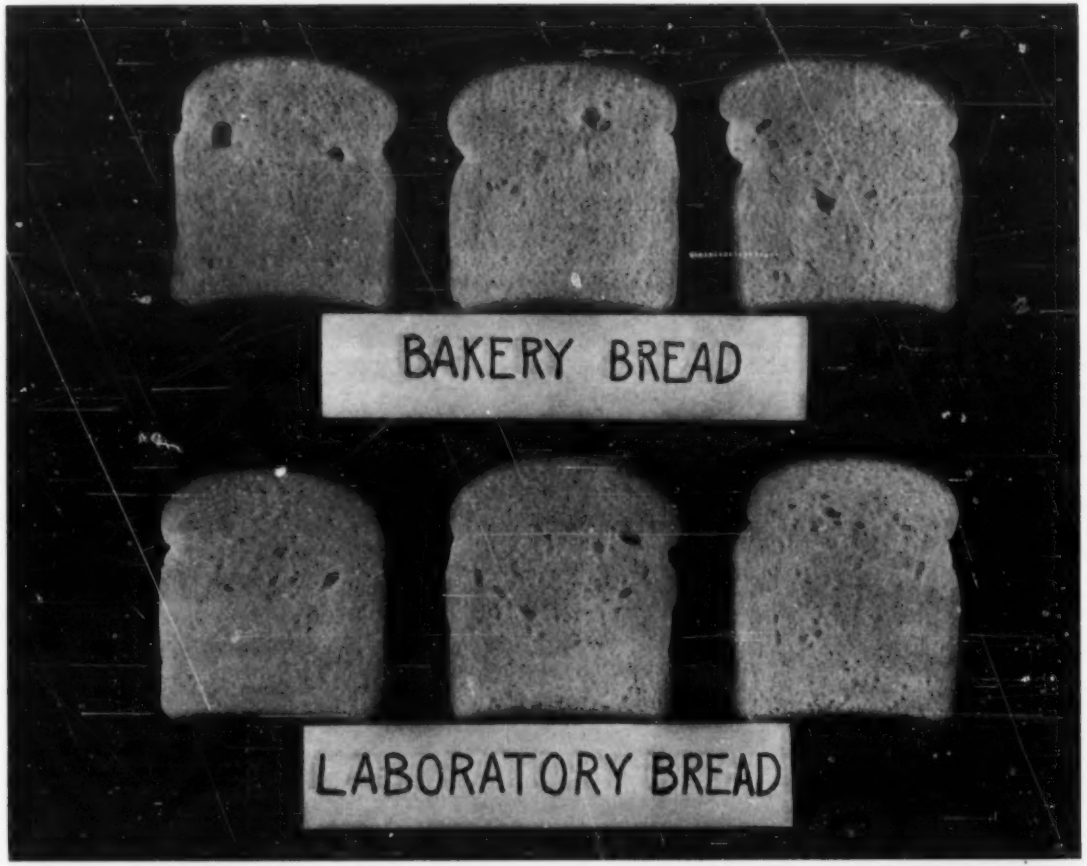

Fig. 8. Bread made from doughs which were handled in the bakery in exactly the same way, after combined mixing and fermentation in the bakery, compared to combined mixing and fermentation in the laboratory. Interior views; bakery and laboratory loaves practically alike.

practically alike.

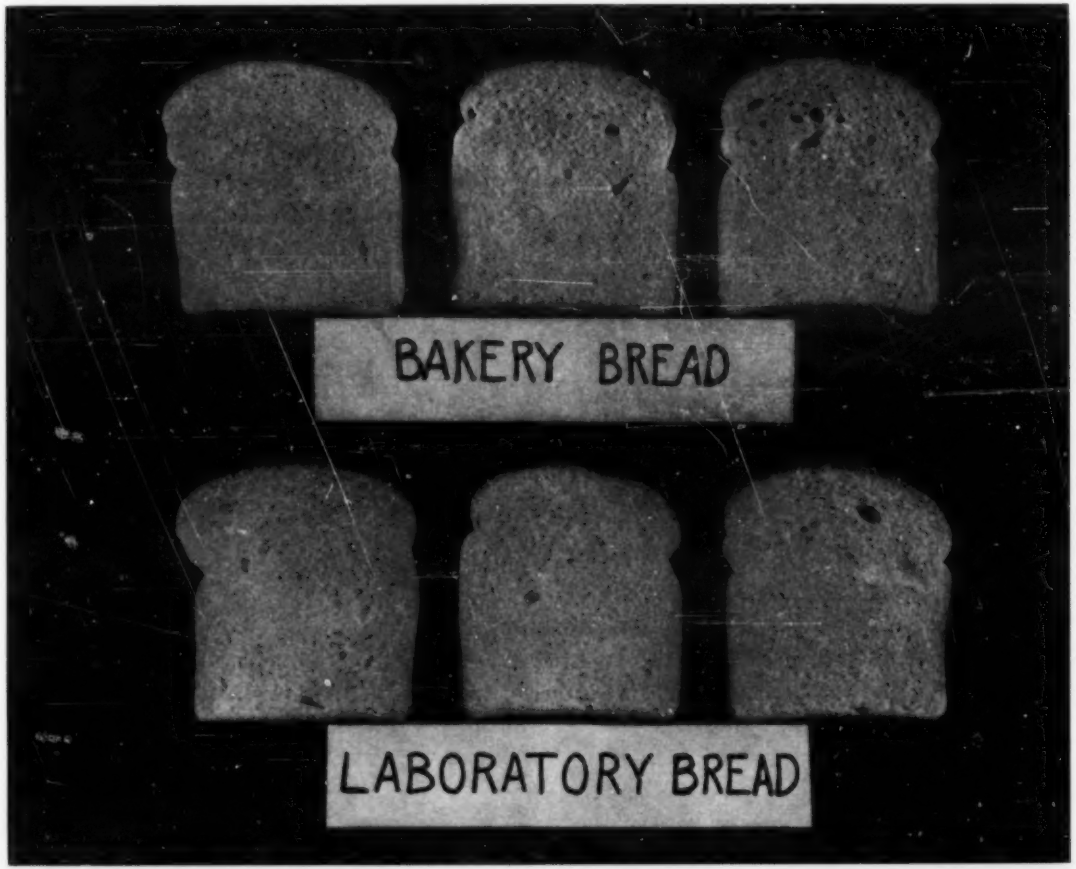

Fig. 9. Bread made from doughs which were handled in the bakery in exactly the same way after combined mixing and fermentation in the bakery, compared to combined mixing and fermentation in the laboratory. Interior views; bakery and laboratory loaves practically alike.

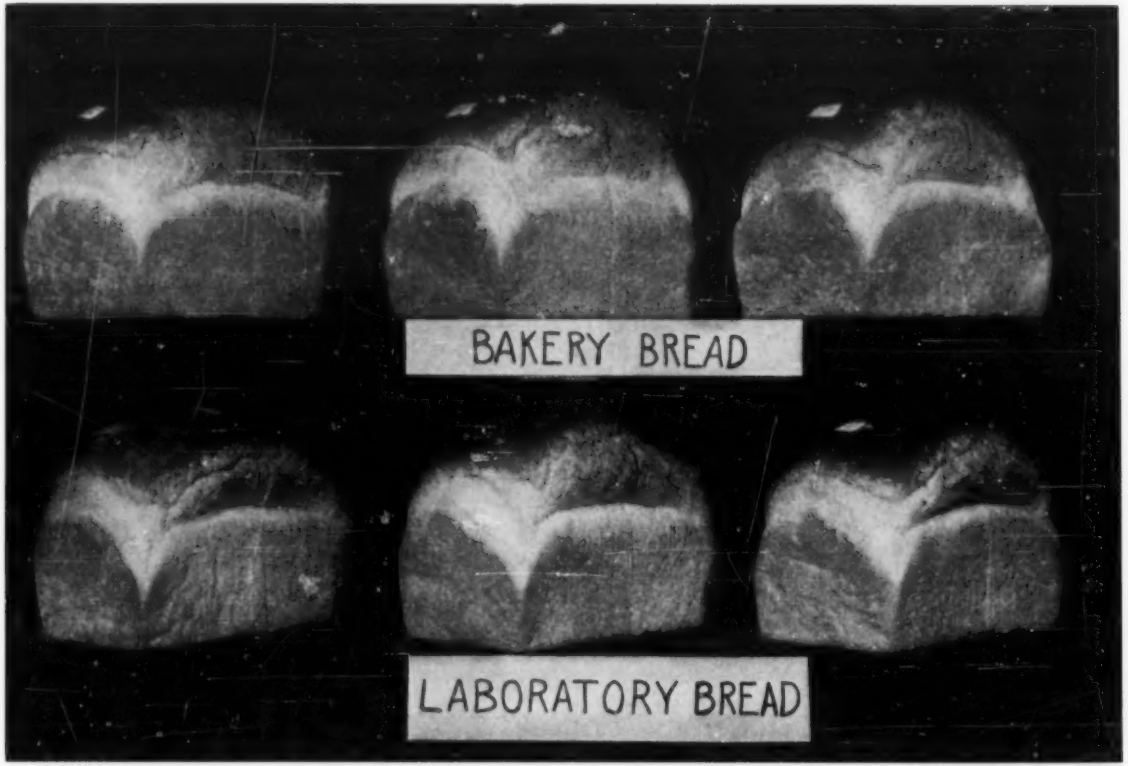

Fig. 10. Exterior views; both doughs handled in laboratory, after combined mixing and fermentation in either bakery or laboratory; loaves practically alike.

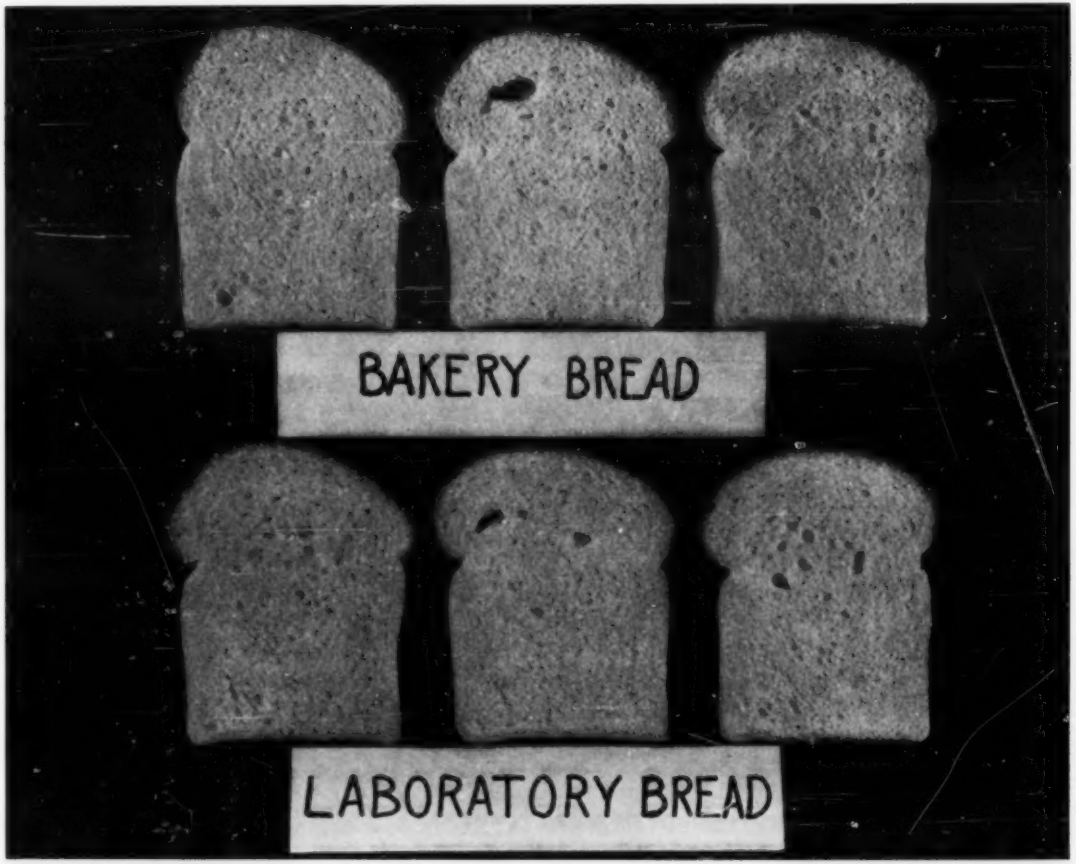

Fig. 11. Interior views; both doughs handled in laboratory, after combined mixing and fermentation in either bakery or laboratory; loaves practically alike.

bread, although both groups looked like satisfactory commercial bread. The bakery loaves were slightly darker in crust color, and somewhat smoother in exterior appearance.

A Brabender-Farinograph test had indicated that to produce the same effect as with smaller doughs, the larger dough would have to be mixed longer in the laboratory mixer. The mixing time was, therefore, increased to 7 minutes in the next experiment, which was, otherwise, a repetition of the first one.

In another experiment, carried on at the same time, some of the same bakery dough was taken to the laboratory after mixing and fermentation in the bakery, and put through the remaining steps in the procedure in the laboratory, compared to a dough which was handled entirely in the laboratory—from mixing to baking. In this experiment, the laboratory molding machine was used, and the loaves were baked in the smaller pans in the rotating tray oven.

The figures for these experiments were as follows:

Handled in Bakery After Mixing and Fermentation

Mixed and fermented in	Mixing time	Dough time	Bench proof	Pan proof	Volume per pound of bread (Average of 7 loaves)
	Min.	*Min.*	*Min.*	*Min.*	*Cc.*
Bakery, at 80° F.	10	143	19	60	2390
Laboratory, at 86° F.	7	133	17	58	2400
Handled in Laboratory After Mixing and Fermentation					
Bakery, at 80° F.	10	140	28	48	2320
Laboratory, at 86° F.	7	140	29	42	2340

In the experiment in which the tests were completed in the bakery, the laboratory mixed and fermented dough again produced darker looking crumb color and older dough characteristics in the resulting bread, as compared to the bakery bread. There were no significant differences in exterior appearance.

In the experiment in which the tests were completed in the laboratory, the indications were the same—the laboratory mixed and fermented dough produced the older looking bread.

These loaves, which were baked in the laboratory in smaller pans, using 17 oz. of dough per pan as against 18 oz. in the larger bakery pans, had finer grain and somewhat different development, but were not dissimilar in general character of texture, compared to the bakery bread baked in the larger bakery pans.

Having had indications in three experiments that the laboratory dough was getting too much fermentation, another experiment was made in which the laboratory fermentation time was reduced.

The figures for this experiment were as follows:

Handled in Bakery After Mixing and Fermentation

Mixed and fermented in	Mixing time	Dough time	Bench proof	Pan proof	Volume per pound of bread (Average of 7 loaves)
	Min.	*Min.*	*Min.*	*Min.*	*Cc.*
Bakery	10	146	19	54	2260
Laboratory	7	115	17	53	2190

This time the laboratory bread appeared to be slightly under fermented; the holes were slightly more rounded and the cell walls not quite as thin as those of the bakery bread. The exteriors of the laboratory bread also indicated a younger dough; the breaks were slightly rough, and the tendency to form shell tops was noticeable.

In the next two experiments the bread produced from doughs mixed and fermented in the laboratory was so much like that of the regular bakery runs, that the loaves could not be distinguished from each other when placed side by side.

The figures were as follows:

Handled in Bakery After Mixing and Fermentation

Mixed and fermented in	Mixing time	Dough time	Bench proof	Pan proof	Volume per pound of bread (Average of 6 loaves)
	Min.	*Min.*	*Min.*	*Min.*	*Cc.*
Experiment (1)					
Bakery	10	150	24	57	2270
Laboratory	7	126	24	54	2360
Experiment (2)					
Bakery	10	145	21	65	2230
Laboratory	7	125	18	63	2260

In crumb color, grain, texture, crust color, and exterior appearance, the laboratory loaves were, in both cases, very much like the bakery loaves. Qualified experts, men experienced in laboratory and large scale bakery production, could not distinguish these loaves when placed side by side.

A useful guide in these tests was dough volume. With the aid of appropriate volume measurements on small dough pieces—fermented

under the same conditions as the larger bakery and laboratory doughs from which they were taken, the latter were fermented to the same volume per unit weight of dough.[3]

In another helpful precaution in the above experiments, in order to give the two doughs identical treatment, 10 lbs. of the large bakery dough was scaled after fermentation, and after the main body of the dough had gone through the divider, and the divider was clean, the two 10-lb. doughs, the bakery and laboratory doughs, were successively put through the divider in exactly the same way.

An experiment similar to the two above, but with the doughs handled in the laboratory after mixing and fermentation and baked in the smaller pans in the rotating tray oven, gave similar results.

The figures were as follows:

Handled in Laboratory After Mixing and Fermentation

Mixed and fermented in	Mixing time	Dough time	Bench proof	Pan proof	Volume per pound of bread (Average of 6 loaves)
	Min.	*Min.*	*Min.*	*Min.*	*Cc.*
Bakery, at 80° F.	10	156	21	60	2220
Laboratory, at 86° F.	7	131	18	50	2230

The laboratory and bakery loaves were exactly alike in internal and external characteristics.

The photographs illustrate the results described in the above experiments.

Summary and Discussion

In a rapid survey of the different steps in the bakery procedure, it was found, in comparative tests using bakery and laboratory methods with the same straight dough formula and ingredients, that laboratory conditions in mixing or fermentation could be so adjusted as to produce results equal to those obtained in the bakery, as indicated by the characteristics of the finished bread. With the exception of hand molding and laboratory baking, this was also found generally true for the other steps in the baking procedure.

Combinations of laboratory mixing and fermentation could be made to reproduce bakery mixing and fermentation in straight dough tests, if after mixing and fermentation, both doughs were handled in exactly the same way in either bakery or laboratory.

[3] For further evidence of amount of fermentation *vs.* fermentation time, see Landis and Freilich Cereal Chem. 11: 396 (1934).

The differences between hand and machine molded bread were so great that there seems to be no choice but the use of machine molding in the laboratory in order to duplicate bakery molding.

The differences between bread baked in the laboratory and bakery ovens were quite marked. This was of course due in part to differences in construction; the large bakery oven had top and bottom heat, steam and dampers, while the small laboratory oven had bottom heat only, no steam, and fans for circulating the air. The use of a small laboratory oven, similar in construction to bakery ovens, would be helpful in a study of the factors involved in any inherent differences between laboratory and large scale baking.

Assuming that all the steps in the baking procedure could be duplicated in the laboratory, they might be classified with respect to their importance in producing differences between bakery and laboratory bread, into major, minor and intermediate factors, as follows:

(*a*) Major factors—mixing, fermentation, baking.
(*b*) Minor factors—rounding, bench proof, pan proof.
(*c*) Intermediate factors—dividing, molding.

This is by no means an exhaustive study of the subject. A great deal of work remains to be done. It may be considered merely an approach to the problem and may serve to indicate along what lines future efforts should be concentrated.

AN EXPERIMENTAL FLOUR MILL FOR 100-GRAM WHEAT SAMPLES [1, 2]

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(Read at the Annual Meeting, June 1935)

In a paper appearing elsewhere in this journal, Geddes and Aitken [3] have reported the results obtained by the use of a small scale experimental milling and baking technique suitable for use with 100 grams of wheat. A general description and the constructional details of the experimental mill employed in these studies are given here.

The mill consists of three units, (1) break rolls, (2) reduction rolls, and (3) bolter. The complete unit is shown in the photograph

[1] Subcommittee report, 1934–35 Committee on the Standardization of Laboratory Baking.
[2] Published as Paper No. 84 of the Associate Committee on Grain Research, National Research Council of Canada, and Dominion Department of Agriculture.
[3] Geddes, W. F., and Aitken, T. R. An experimental milling and baking technique requiring 100 grams wheat. Cereal Chem. 12: 696–707.

in Figure 1, while the break rolls with the feed hopper thrown back are shown in Figure 2. The break and reduction rolls are modelled upon the lines of the standard Allis-Chalmers experimental mill, the

Fig. 1. Small scale experimental mill (complete unit).

roll diameters and speeds being identical, but with the surfaces reduced to an effective length of one inch, as compared with six inches for the standard Allis-Chalmers. The bolter is remodelled from a standard Allis unit, the sieve area and depth being scaled down to suit the smaller sample involved.

Constructional Details

A detailed drawing of the reduction roll unit is given in Figure 3; the break roll unit is identical to this with the exception that the rolls are corrugated and the scraper omitted. The main frames and tension arms are machined from gray iron castings, the details and dimensions of which are shown in Figure 4, the bases and hoppers being built up from wood. The feed hoppers are hinged as shown, in order to facilitate cleaning.

In designing a mill to operate on 100-gram samples, it is obviously essential to reduce loss of sample to a minimum; for this reason the mill itself is mounted directly upon a heavy wooden base, into which is fitted a drawer for the reception of the mill products.

The rolls are 6 inches in diameter, machined from locomotive crank-pin steel and pressed on to the shafts. The break rolls are corrugated with the standard Allis cut, 18 per inch on the fast, 16 per inch on the slow, and are arranged to operate "dull" to "dull." The reduction rolls are machined very slightly oversize, the surfaces hardened by carbonizing and finally ground true. The actual roll

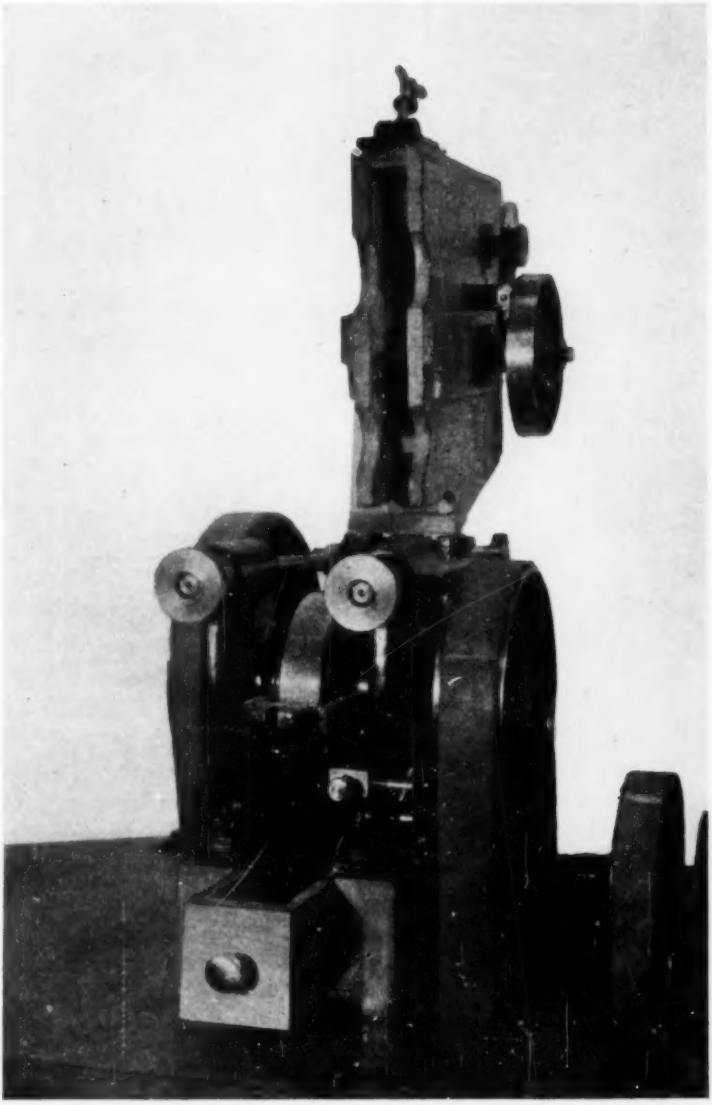

Fig. 2. Break roll unit.

width is 1¾ inches, but the effective working face is reduced to approximately one inch by means of ⅜ inch thick wooden inserts fitted to the inside of the lower portion of the feed hopper. Bronze bearings are employed throughout, lubrication being obtained with oil cups and suitable channels. A departure from conventional practice is made in the arrangement of the tension arms. In the usual type of labora-

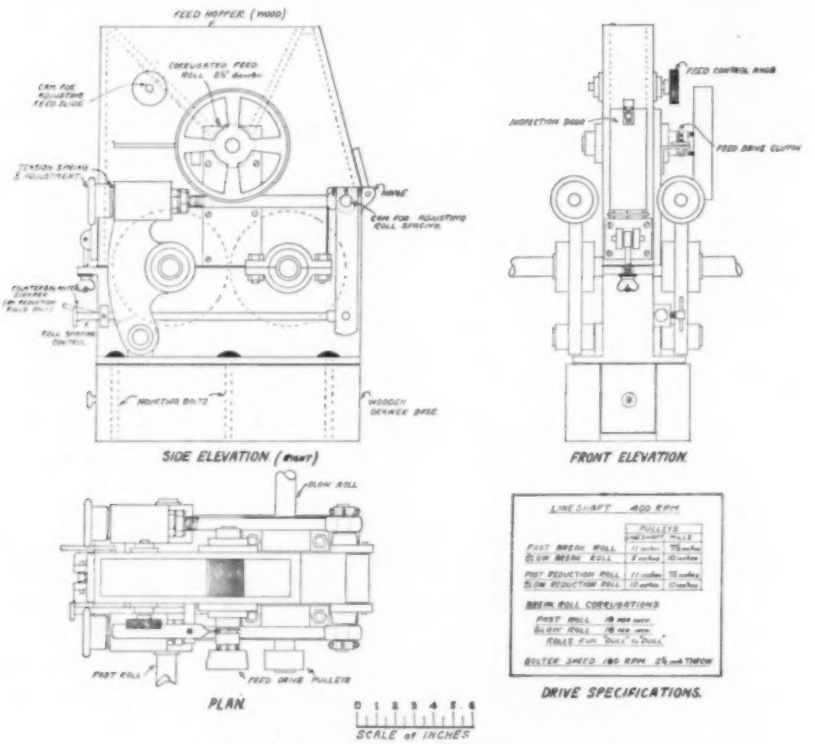

Fig. 3. Detail of reduction roll unit and drive specifications.

tory mill, these arms are pivoted independently, in order to adjust the parallelism of the rolls. This would be an exceedingly difficult adjustment to make in the case of the 100-gram mill, and both tension arms are therefore permanently attached to a single pivot, parallelism being established at the time the mill is assembled. Roll spacing is accomplished by means of a lever-operated eccentric in the usual manner.

A counterbalanced scraper is fitted to the fast smooth roll to remove any particles which may adhere to the surface.

The feed mechanism follows the prototype quite closely, employing an eccentric-operated slide and a 2½ inch feed roll belt driven from the

slow roll. The feed is disconnected when desired by means of a simple collar clutch.

The bolter consists of a wooden casing $6\frac{1}{2}'' \times 6\frac{1}{2}'' \times 8\frac{1}{4}''$ with a wall thickness of $\frac{3}{8}''$ capable of holding four sieves, and a metal drawer fitted into the lower portion. The sieves are $5\frac{7}{16}''$ square

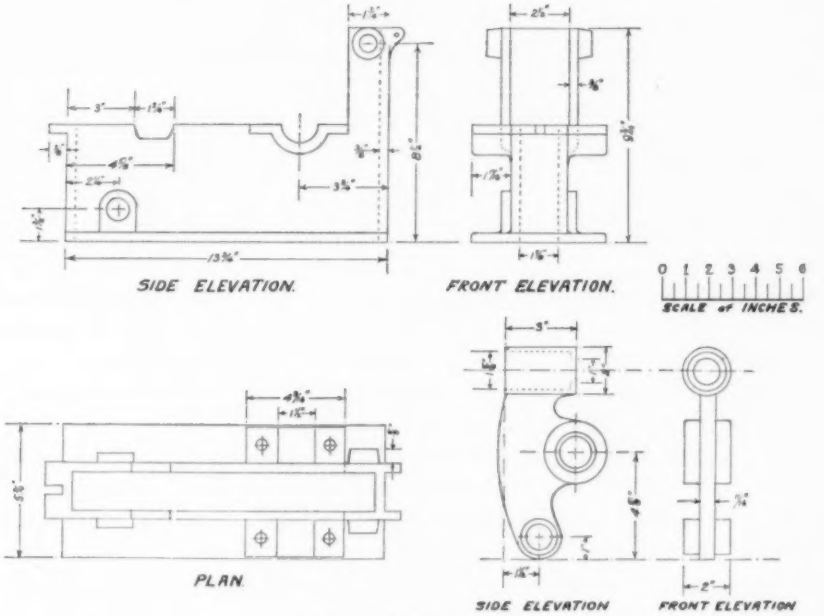

Fig. 4. Detail of castings.

(outside dimensions) and $1\frac{1}{2}''$ deep, Nos. 18-W, 40-GG, 70-GG, and 10-XX being used for the breaks, and Nos. 20-W, 40-GG, 50-GG, 70-GG, and 10-XX for the reductions; No. 40-GG replaces No. 50-GG in the fourth reduction. The entire assembly is mounted upon a wooden platform which replaces the sieve box of a standard Allis-Chalmers experimental bolter.

The necessity of a separate power supply is obviated by mounting the two stands and bolter on an extension built on to the end of the standard Allis-Chalmers experimental mill unit, the power being derived by extending the line shaft.

Acknowledgments

The authors are indebted to I. S. Snider, Experimental Miller, Grain Research Laboratory, and to Carl Hansen for valuable assistance in designing and constructing the experimental mill. They are also indebted to D. S. Binnington, Research Assistant, National Research Council, who made the detailed drawings.

AN EXPERIMENTAL MILLING AND BAKING TECHNIQUE REQUIRING 100 GRAMS WHEAT [1, 2]

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(Read at the Annual Meeting, June 1935)

One of the major problems confronting the originators of new varieties of wheat is the application of suitable quality tests to small quantities of material, permitting the selecting of promising strains and the discarding of unsatisfactory lines early in the plant-breeding program. In the F_3 and subsequent generations, until new varieties are being increased for plot tests, the quantity of surplus seed of each family usually varies from approximately 100 to 300 grams, so that several years must elapse before sufficient seed is available for making the standard milling and baking tests.

The wheat-meal fermentation time test developed by Cutler and Worzella (1931, 1933) in America, and Pelshenke (1933) in Germany aroused a great deal of interest as a possible means of evaluating wheat strength. The test is simple, requires only small quantities of material and is based on the resistance of a fermenting dough, made from medium finely ground whole wheat meal, to disintegration in water, the time factor involved being regarded as a measure of baking strength.

Bayfield (1935) has recently reviewed the results obtained by numerous investigators in evaluating wheats of various classes by this test, and it would appear that while it is of some value in roughly classifying wheats on a strength basis, it only proves entirely successful with samples of such widely varying strength as could be readily differentiated by means of the protein test. The correlations found between the "disintegration time" in the wheat-meal test and baking strength as determined by actual baking tests are not sufficiently high to consider the method a satisfactory substitute for experimental milling and baking tests.

In producing new wheat varieties, the plant breeder endeavours not only to combine desirable agronomic characteristics with high baking strength but also to produce wheats which yield flour of low

[1] Subcommittee report, 1934–35 Committee on the Standardization of Laboratory Baking.
[2] Published as Paper No. 85 of the Associate Committee on Grain Research, National Research Council of Canada, and Dominion Department of Agriculture.

pigment content. Goulden, Geddes, and Whiteside (1934) have studied the relation between the carotenoid pigment content of ground whole wheat and straight grade flour milled therefrom and, while the results indicated that tests on whole wheat might be of considerable value to plant breeders for eliminating highly pigmented varieties in the early stages, the correlations between wheat and flour carotene were not sufficiently high to permit an entirely satisfactory prediction of the carotene content of the flour from tests on wheat.

The need for suitable milling equipment which would permit the production of flour similar in character to that obtained from the regular experimental mill, and a baking procedure which would yield miniature loaves, employing only small samples of wheat and flour, prompted the attempt to develop the small scale laboratory milling and baking technique described in this paper.

Experimental

Milling Tests

A description of an experimental mill for the production of flour from small samples of wheat is reported elsewhere in this journal by Geddes and Frisell (1935). Preliminary tests undertaken with a view to developing a suitable milling technique showed that no major changes in the flow sheet employed in this laboratory for the regular three-stand (2 sets of corrugated rolls, Nos. 16 and 24, and 1 set of smooth rolls) Allis-Chalmers experimental mill as described by Birchard (1932) were necessary. The customary five breaks yielded mill products which closely corresponded in appearance to those produced by the Allis-Chalmers mill, excepting that the bran was somewhat smaller in size, owing to the use of only one set of corrugated rolls. Accordingly, a series of wheats of widely varying milling and baking strength, listed in Table I, was milled on the miniature mill and on the Allis-Chalmers experimental mill, employing 150 grams and 2000 grams wheat respectively, one milling only of each sample being made in each case. While the miniature mill is designed to produce flour from wheat samples as small as 100 grams, in these original studies 150 grams was used so that sufficient flour would be available for determinations of protein, ash, carotene, and diastatic activity.

Owing to differences in kernel texture of the wheats employed, it became necessary to mill the samples at different moisture levels, 14% being used for the Australian, Austrian, English, French, German, Hungarian, and Italian; 15% for the Argentinian, United States (Kansas), Russian, and Canadian of the first five grades; and 16% for the Canadian grades Nos. Five and Six.

TABLE I

ORIGIN, WEIGHT PER BUSHEL, AND PROTEIN CONTENT OF WHEATS

Reference number	Sample [1]	Weight per bushel (Cleaned wheat) [2]	Protein content (N × 5.7) [3]
		Lbs.	%
1	Standard 1 Northern	62	14.7
2	Standard 2 Northern	61¾	14.7
3	Standard 3 Northern	61¾	14.5
4	Winnipeg Average 1 Northern	63½	14.4
5	Standard 1 Hard	63½	14.3
6	Winnipeg Average 1 Hard	65¼	14.1
7	Winnipeg Average 2 Northern	63	14.0
8	Winnipeg Average 1 Northern	64	13.5
9	Standard 4 Northern	64	13.2
10	Russian	65¼	13.2
11	Winnipeg Average 3 Northern	63½	13.1
12	Standard No. 6	61½	12.7
13	Argentinian (Up River)	62	12.7
14	Winnipeg Average 4 Northern	63¼	12.6
15	Kansas Red Winter	66	12.6
16	Hungarian	65½	12.6
17	Standard No. 5	62¼	12.4
18	Winnipeg Average 4 Northern	64	12.3
19	Winnipeg Average No. 5	62¼	11.8
20	Winnipeg Average No. 6	60½	11.6
21	Argentinian (Rosafé)	64	11.6
22	Argentinian (type unknown)	62¾	11.1
23	Australian	61	10.8
24	German	63	10.3
25	French	63½	10.1
26	Italian	61	9.7
27	Austrian	62	9.6
28	English	64	9.4

[1] The Standards and Winnipeg Averages are all Canadian hard red spring wheats.
[2] Imperial bushel.
[3] 13.5% moisture basis.

To control the end point of the millings, the customary standard samples of shorts and feed flour used in this laboratory for routine milling tests were employed, extraction of flour from the shorts being continued until the latter matched the standard in color; similarly reduction of the feed flour was carried on until the residual feed flour corresponded in colour with the standard.

The flour yields, total products recovered, and the results of chemical analyses are given in Table II. Nitrogen was determined by the Kjeldahl-Gunning-Arnold procedure, ash by the standard A. A. C. C. procedure, diastatic activity by the method outlined by Blish and Sandstedt (1933), and carotene by the spectrophotometric method of Ferrari (1933), using the naphtha-alcohol solvent of the characteristics described by Geddes *et al.* (1934).

The statistical constants for the data given in Table II are summarized in Table III. In order to secure a measure of the significance

TABLE II

Milling and Miscellaneous Data

Reference number	Sample	Milling data: Flour yield, Allis mill	Milling data: Flour yield, 100-g. mill	Milling data: Milling loss, Allis mill	Milling data: Milling loss, 100-g. mill	Analyses of flours (13.5% moisture basis): Protein (N×5.7), Allis mill	Protein (N×5.7), 100-g. mill	Ash, Allis mill	Ash, 100-g. mill	Carotene, Allis mill	Carotene, 100-g. mill	Diastatic activity[1], Allis mill	Diastatic activity[1], 100-g. mill
		%	%	%	%	%	%	%	%	*p.p.m.*	*p.p.m.*	(*mg. maltose per 10 g. flour*)	
1	Standard 1 Northern	70.0	67.3	1.0	4.1	14.0	13.8	0.46	0.47	1.82	2.02	105	112
2	Standard 2 Northern	69.7	65.3	0.9	4.2	13.8	13.6	0.48	0.49	2.01	2.10	126	124
3	Standard 3 Northern	69.8	65.3	1.1	4.5	13.6	13.8	0.48	0.50	2.04	2.14	139	142
4	Winnipeg Average 1 Northern	71.0	66.6	0.6	2.9	13.6	13.4	0.46	0.54	1.72	2.00	125	133
5	Standard 1 Hard	70.4	66.6	1.1	4.2	13.2	13.2	0.41	0.46	1.67	1.89	96	118
6	Winnipeg Average 1 Hard	72.1	66.0	1.4	5.3	13.5	13.0	0.43	0.45	1.64	1.81	117	128
7	Winnipeg Average 2 Northern	70.5	66.6	1.2	4.8	13.2	13.2	0.46	0.49	2.01	2.04	124	108
8	Winnipeg Average 1 Northern	71.9	66.6	0.9	4.8	12.9	12.9	0.45	0.49	1.87	2.00	105	131
9	Standard 4 Northern	69.2	64.0	0.9	5.4	12.2	12.3	0.46	0.50	1.94	2.02	178	205
10	Russian	70.9	67.3	1.1	2.1	12.3	12.4	0.53	0.56	1.55	1.68	133	130
11	Winnipeg Average 3 Northern	70.6	66.2	1.1	3.2	12.8	12.6	0.46	0.53	2.01	2.02	153	140
12	Standard No. 6	62.8	58.6	0.7	1.2	11.4	11.3	0.53	0.58	1.64	1.94	259	357
13	Argentinian	68.0	65.3	1.5	2.8	12.8	12.3	0.51	0.51	1.83	1.98	73	78
14	Winnipeg Average 4 Northern	69.4	65.3	1.3	4.1	11.7	11.9	0.50	0.53	1.89	2.08	188	176
15	Kansas Red Winter	72.0	68.0	1.0	2.7	11.6	11.8	0.47	0.57	1.62	1.78	102	118
16	Hungarian	70.8	68.0	0.5	2.2	11.8	11.8	0.43	0.49	1.76	1.93	121	111
17	Standard No. 5	66.4	62.6	0.3	0.9	11.3	11.1	0.47	0.52	1.80	2.01	243	275
18	Winnipeg Average 4 Northern	69.6	66.0	0.9	2.1	11.7	11.7	0.49	0.52	1.88	2.14	165	182
19	Winnipeg Average No. 5	66.5	60.6	0.8	2.2	11.0	11.2	0.51	0.56	1.88	2.11	233	252
20	Winnipeg Average No. 6	63.6	59.0	1.0	1.1	10.7	10.9	0.55	0.59	1.82	2.04	262	328
21	Argentinian	69.2	66.0	1.6	4.1	10.7	10.7	0.50	0.55	1.68	1.70	89	80
22	Argentinian	67.3	65.3	1.0	3.4	10.6	10.4	0.51	0.52	1.94	2.10	110	108
23	Australian	68.0	65.3	1.6	3.4	9.6	9.3	0.41	0.44	2.10	2.14	94	92
24	German	70.6	66.6	1.0	4.2	9.3	9.0	0.49	0.49	2.13	2.20	78	66
25	French	66.3	65.3	1.1	3.4	8.9	9.0	0.47	0.51	1.88	1.92	92	83
26	Italian	67.2	65.3	1.2	2.1	8.9	9.0	0.49	0.49	1.30	1.35	59	72
27	Austrian	69.6	66.6	1.1	2.9	8.8	9.3	0.53	0.54	2.61	2.86	110	125
28	English	70.0	66.6	1.1	5.4	8.0	7.8	0.40	0.46	2.24	2.27	82	88

[1] Means.

TABLE III

Statistical Constants for Milling Data and Chemical Determinations on Flours

Determination	Means: Allis mill	Means: 100-g. mill	Means: *F* value[1]	Means: 5% point (approx.)	Standard deviations: Allis mill	Standard deviations: 100-g. mill	Coefficients of variability: Allis mill	Coefficients of variability: 100-g. mill	Correlation coefficients: (Allis ×100 g.)[1]
Flour yield, %	69.000	65.300	35.62	4.02	2.340	2.380	3.40	3.60	.878
Milling loss, %	1.000	3.400	—	—	—	—	—	—	—
Protein content, %	11.570	11.520	.01	254.00	1.730	1.690	14.90	14.60	.991
Ash content, %	.476	.512	12.04	4.02	.036	.039	7.50	7.80	.797
Carotene content, p.p.m.	1.867	2.010	4.65	4.02	.245	.249	13.12	12.39	.939
Diastatic activity	134.300	145.100	.37	254.00	56.590	74.800	42.10	51.60	.968

[1] Value of r at 5% point = .365.

of the differences between the mean values for corresponding determinations by the two milling procedures, the data were submitted to

variance analyses, the results of which are summarized by the F values for the ratio of the variance between milling methods and that for within milling methods. When the F values exceed their 5% points, the corresponding means may be regarded as significantly different.

It will be noted that the 100-gram mill gives a lower yield of flour and a lower recovery of total products than the Allis mill, the resultant flour being equal to that from the Allis mill in protein content but significantly higher in ash and carotene. While the mean diastatic activity value is also higher for the 100-gram mill, the difference is not statistically significant, owing to the fact that the small mill does not consistently yield flours of higher diastatic activity.

The mean milling loss amounted to 3.4% on the 100-gram mill as compared with 1.0% on the Allis-Chalmers, the higher loss being unavoidable owing to the fact that the small quantities of stock employed require the same series of manipulations as those of the larger mill, a 1.5 gram loss corresponding to 1.0% as compared with only 0.08% for the Allis mill. The lower flour yields obtained on the 100-gram mill are due very largely to the higher milling loss, since the greater portion of the unrecovered material is flour. The higher values for ash and carotene indicate greater contamination of the flour obtained in the 100-gram mill with the mill offals; the correlations between the results of corresponding determinations in the two mills are, however, quite high. This is of particular importance in regard to carotene, since heretofore there has been no entirely satisfactory means available to plant breeders for the elimination of wheat hybrids in early generations which would yield flours of undesirably high yellow pigmentation. In the absence of a suitable method for producing flour from small samples of wheat, Goulden, Geddes, and Whiteside (1934) studied the relation between the carotene content of ground whole wheat and straight grade flour milled therefrom, in order to determine whether the degree of association was sufficiently high to permit the prediction of flour carotene from a knowledge of wheat carotene. The highest correlation obtained was .702 as compared with .939 for the relation between the carotene content of flours produced in the Allis and 100-gram mills.

Since for a linear relationship the standard error of prediction of a single predicted observation obtained by having recourse to the regression equation is defined by $\sqrt{1-r^2}$ times the standard deviation of the dependent variable, the data obtained in this study indicate that the carotene content of the flour obtained by the regular experimental milling procedure may be predicted with a standard error of only 0.08 p.p.m. This may be regarded as highly satisfactory. As

carotene determinations on ground whole wheat require uniform and fine grinding of the samples, Goulden *et al.* employed a Wiley mill, fitted with an 0.5 mm. mesh sieve which requires a time of approximately 20 minutes to grind a 50-gram sample. The average time to complete a milling of 150 grams wheat including the recording of the results was also only about 20 minutes, so that with the aid of this small mill a much more satisfactory product for carotene studies may be obtained in the same time.

Baking Tests

In view of the fact that 150 grams wheat yield only from 85 to 100 grams long patent flour, it is impossible to conduct baking tests employing the quantity of flour specified in the A. A. C. C. official baking test, and it was necessary to bake 25-gram flour doughs; a few preliminary tests reported by Geddes and Sibbitt (1933) indicated that such a procedure might satisfactorily differentiate between flours on a loaf volume basis.

As a 25-gram flour dough cannot be mixed in either the Hobart or Hobart-Swanson mixers, it was necessary to mix 50 grams flour at the one time and divide the dough into two equal parts immediately after mixing, from which loaves were baked. Special baking tins, constructed of 2XX tin, were secured, the inside dimensions of which were: top, 7.1 × 4.4 cm.; bottom, 5.9 × 3.3 cm.; height, 3.2 cm., so that these pans are similar to the standard low-form tins, the dimensions being scaled down to maintain the same ratio of dough weight to pan volume as the official "low-form" tin. For these initial experiments, the small loaves were measured by means of rape seed displacement in a small box of known cubic capacity, while the "100-gram" loaves were measured in a device similar to that described by Geddes and Binnington (1928). With these exceptions, the baking procedure followed was the Official A. A. C. C. Procedure (Geddes, 1934), the baking formula being the malt-phosphate-bromate [basic plus 0.3% diastatic malt (200° Lintner), 0.1% $NH_4H_2PO_4$ and 0.001% $KBrO_3$]. Experience has shown that this formula gives a wide differentiation between the loaf volumes of experimentally milled flours.

As the surfaces of the cut loaves from the 25-gram doughs were too small to satisfactorily score for crumb color and crumb texture, the only baking data recorded were loaf volume and external appearance.

To obtain information regarding the comparative baking strength of the flours milled from corresponding wheats on the two mills, the flours from the Allis-Chalmers mill were baked using both the 100-gram and 25-gram flour doughs, and those from the small mill by the

25-gram dough only. In all three series, duplicate loaves were baked from each sample.

The mean loaf volumes for the duplicate bakings for each series are recorded in Table IV, and in Figure 1 the means for the two series

TABLE IV

MEAN LOAF VOLUME DATA

Reference number	Sample	Allis mill		100-gram mill
		100-g. loaf	25-g. loaf	25-g. loaf
		Cc.	*Cc.*	*Cc.*
1	Standard 1 Northern	970	202	201
2	Standard 2 Northern	915	196	190
3	Standard 3 Northern	930	182	199
4	Winnipeg Average 1 Northern	870	188	194
5	Standard 1 Hard	855	182	186
6	Winnipeg Average 1 Hard	938	202	194
7	Winnipeg Average 2 Northern	888	194	192
8	Winnipeg Average 1 Northern	902	194	190
9	Standard 4 Northern	805	169	177
10	Russian	718	148	152
11	Winnipeg Average 3 Northern	870	183	185
12	Standard No. 6	520	108	115
13	Argentinian	602	120	115
14	Winnipeg Average 4 Northern	752	160	161
15	Kansas Red Winter	785	160	162
16	Hungarian	682	138	148
17	Standard No. 5	595	130	130
18	Winnipeg Average 4 Northern	770	148	154
19	Winnipeg Average No. 5	648	141	140
20	Winnipeg Average No. 6	522	109	120
21	Argentinian	610	120	114
22	Argentinian	670	135	136
23	Australian	640	134	131
24	German	558	120	112
25	French	530	112	110
26	Italian	520	109	99
27	Austrian	570	112	122
28	English	460	96	93

of 25-gram bakes are plotted against the corresponding values for the 100-gram loaves.

Analyses of variance were conducted on the individual loaf volumes obtained by the three series of bakings, and the results of these, together with the other statistics computed, are given in Table V.

From the F values expressing the ratio of the variance for between flours to that for between duplicates, it will be noted that the differentiation of wheat strength, as measured by loaf volume, is greatest by the 100-gram bake. The coefficients of variability of the mean loaf volumes, however, show that the relative spread between flours is quite similar for the three series, while the experimental error ex-

pressed in terms of coefficient of variability, as would be expected, is somewhat higher for the small mill loaves, and this is responsible for the decreased precision in differentiating between flours by the small scale technique. The correlations between the mean loaf volumes for the various combinations shown in Table V are all significant and of a very high order of magnitude and indicate that the small scale milling

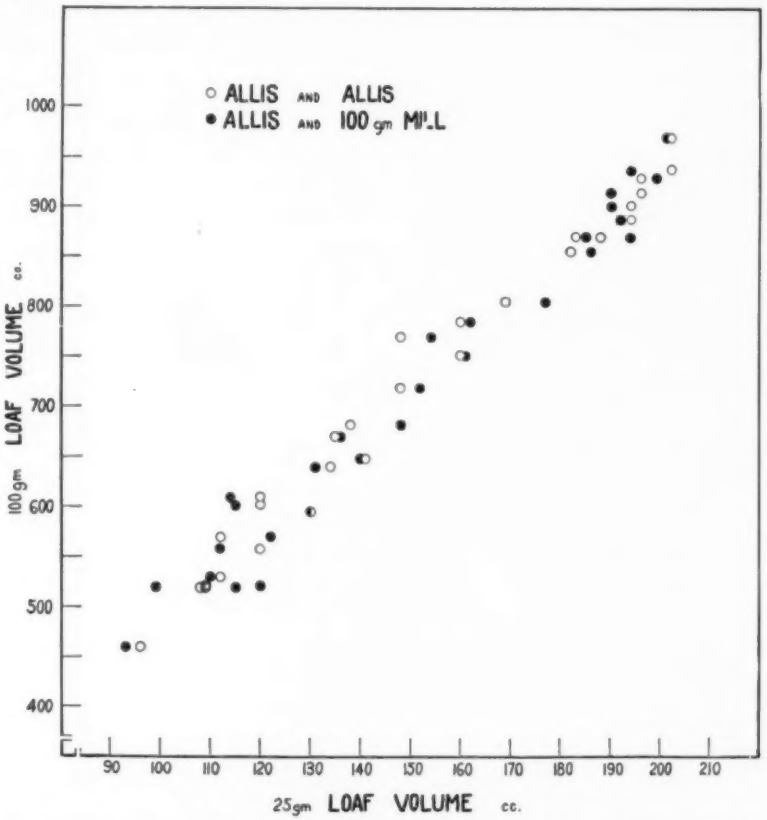

Fig. 1. Means for the two series of 25-gram bakes plotted against the corresponding values for the 100-gram loaves.

and baking techniques give similar results to the regular experimental procedures. The external characteristics of the "100-gram" and "25-gram" loaves baked from corresponding flours were strikingly similar, the small loaves being miniatures of the large loaves in almost every particular. By having recourse to the regression equation, the mean loaf volume of duplicate 100-gram bakings of one flour produced on the Allis-Chalmers mill in this series may be predicted with a standard error of approximately 30 cc., which may be regarded as quite satisfactory.

TABLE V

STATISTICAL CONSTANTS FOR LOAF VOLUME DATA

Variance due to	Degrees of freedom	Variance	*F* value	5% point	Standard deviation	Coefficient of variability
Flours Milled on Allis-Chalmers Mill						
(*a*) 100-gram bake						
Differences between flours	27	48,895.31	405.64	1.90	156.36	21.79
Differences between duplicates	28	120.54	—	—	10.98	1.53
(*b*) 25-gram bake						
Differences between flours	27	2314.50	258.31	1.90	34.02	22.72
Differences between duplicates	28	8.96	—	—	2.99	2.00
Flour Milled on Small Scale Experimental Mill—25-gram Bake						
Differences between flours	27	2421.54	253.47	1.90	34.80	23.34
Differences between duplicates	28	9.55	—	—	3.09	2.07

Mean Loaf Volumes

	Cc.
Flours milled on Allis-Chalmers mill:	
(*a*) 100-gram bake	717.7
(*b*) 25-gram bake	149.8
Flours milled on small scale mill—25-gram bake	149.1

Simple Correlation Coefficients

	r
Large Mill—100-gram bake × 25-gram bake	.970
Large Mill—100-gram bake × small mill 25-gram bake	.984
Large Mill— 25-gram bake × small mill 25-gram bake	.982
At 5% point	$r = .365$

Linear Regression Coefficient

Large mill—100-gram loaf$^{(x)}$ on small mill 25-gram loaf$^{(y)}$ $B_{xy} = 4.413$

Linear Regression Equation

Estimation of 100-gram loaf volume (x) (large mill) from 25-gram loaf volume (y) (small mill)

$$x = 59.75 + 4.413y$$

Standard error of prediction = 29.6 cc.

Discussion

The results obtained on the series of 28 wheats of widely differing baking strength indicate that the small scale milling and baking technique requiring a minimum of 100 grams wheat would be of considerable value to plant breeders and others requiring information as to the milling and baking characteristics of wheat when only small quantities of material are available. Differences in the milling behaviour of the various types of wheat employed in this study were as readily detected on the small as on the regular sized Allis-Chalmers mill. In experimental work of this nature one is not primarily interested in flour yield, since this factor is closely related to weight per bushel and soundness of the wheat. One of the outstanding features of the small scale mill is the speed with which millings may be carried out, and with less fatigue to the operator, since the whole unit occupies a comparatively small area. In testing early generations of wheat hybrids for the purpose of eliminating inferior strains, a large volume of tests is necessary and the rapidity with which flour may be produced for carotene or other tests by the small scale mill is a distinct advantage.

While the higher relative experimental error of the small scale baking procedure decreases the differentiation between flours, it is sufficiently precise for the purpose of selecting definitely inferior or superior wheats of the same class. This is shown by the results on the 16 hard red spring wheats originating in Canada, included in the present study. In addition, the efficacy of the method has been tested by applying it to a series of six named varieties of Canadian hard red spring wheat in comparison with the regular laboratory procedures. While the differentiation in loaf volume between the varieties was somewhat less for the former, the wheats were classified in precisely the same order as by the latter method and in line with their known commercial rating.

The inability to satisfactorily score the 25-gram loaves for crumb color and crumb texture does not detract from the value of the small scale baking test, since, in testing wheat strength, one is primarily interested in securing a measure of potential strength and for this purpose it is necessary to employ baking formulas which in the instance of strong wheats, produce large loaves without regard to texture. As is the case with crumb texture, crumb color is influenced by loaf volume and a better indication of the relative crumb colors which would be obtained in loaves of corresponding volumes may be obtained from a knowledge of the pigment content of the flour.

Summary

Twenty-eight wheats including samples of widely varying protein content and baking strength were milled to yield on the regular sized Allis-Chalmers and also on the small scale experimental mill built to process a minimum of 100 grams. The Allis mill flours were baked by the standard 100-gram and also by a 25-gram procedure, while the small mill flours were baked by the 25-gram method only, the malt-phosphate-bromate formula being employed in each case.

The miniature mill gave a lower yield of flour and a lower recovery of total products, but the resultant flours were equal to those from the Allis mill in protein content and significantly higher in ash and carotene contents. While the diastatic activities of the small mill flours were, on the whole, also higher than those of the Allis mill, the differences were not statistically significant due to the fact that the former mill did not consistently yield flours of higher diastatic value. The milling loss by the small mill was 3.4% as compared with 1.0% by the Allis mill. Correlation coefficients for flour yield and for the protein, ash, carotene and diastatic activity data on the flours produced by the two milling procedures were .878, .991, .797, .939, and .968 respectively.

The average time required to complete a milling on the miniature mill is about 20 minutes, which is not longer than the time required to grind wheat on the Wiley mill to a satisfactory fineness for carotene determinations.

The differentiation of wheat strength is greater by the 100-gram baking procedure, but the relative spread between flours is quite similar for the three series of samples. As would be expected, the experimental error is somewhat higher for the miniature mill 25-gram loaves, which is responsible for the decreased precision in differentiating between flours by the small scale technique. The correlations between the mean loaf volumes obtained by the different methods were: between the 100-gram and 25-gram Allis mill loaves .970, between the 100-gram Allis and 25-gram miniature mill .984, and between the two sets of 25-gram loaves .982. The external characteristics of the "100 gram" and "25 gram" loaves baked from corresponding flours were strikingly similar, the small loaves being miniatures of the large ones in almost every particular.

There is no evidence to show that the small scale milling procedure produces flour of different baking behaviour by the formula employed from that obtained on the regular sized Allis mill when judged by the 25-gram and 100-gram baking methods, respectively. The regression equation shows that the mean loaf volume of duplicate 100-gram loaves from flour produced on the Allis mill, in this series, may be predicted with a standard error of approximately 30 cc.

Acknowledgments

The authors are indebted to I. S. Snider, Experimental Miller, and M. H. Fisher, Experimental Baker, both of the Grain Research Laboratory, who conducted the milling and baking tests connected with these studies.

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CAROTENOID PIGMENTS IN WHEAT WITH SPECIAL REFERENCE TO VARIETIES AND STRAINS [1]

W. W. Worzella and G. H. Cutler [2]

(Received for publication June 24, 1935)

In the process of developing new and suitable varieties of wheat, the plant breeder is charged with the responsibility of combining such characters as are of direct importance to the interests of the farmer and consumer. Among these, the colour of flour a wheat is capable of producing seems important, since it greatly influences its market value and utility. According to recent studies, the amount of carotenoid pigments in wheat is closely related to colour of the resulting products. This being true, the question arises, is it possible for the wheat breeder to produce new wheats of suitable carotenoid content? The purpose of this study was to determine the possibilities of breeding new wheat varieties possessing the desired amount of carotenoid pigments. This paper reports a study of the quantitative amount of carotenoid pigments in different varieties and hybrid strains of wheat. Finely ground whole wheat meal was used in all experiments.

Review of Literature

Whiteside (1931) found a high correlation between the carotenoid content of finely ground whole wheat meal and patent flour milled from the same wheat. The carotenoid content of the patent flour also correlated with the crumb colour score of the bread.

Goulden, Geddes, and Whiteside (1934) obtained a coefficient of + 0.7225 between carotene in flour and whole wheat meal, and — 0.4951 between whole wheat meal and crumb colour score, when using naphtha-alcohol solvent.

Whiteside, Edgar, and Goulden (1934) show that two factors have a bearing on the carotene values obtained, namely, environmental effects and inherent varietal characteristics.

Materials and Methods

The experiments here reported were conducted largely with soft wheats made up of 29 commercial varieties and 72 hybrid strains grown

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at Lafayette, Indiana, during 1932–33 and 1933–34. Each variety or hybrid strain was grown in rod-row plots replicated 4 times, and a composite sample of each used in this experiment.

The carotenoid pigmentation, expressed as carotene in parts per million of finely ground whole wheat meal, was determined spectrophotometrically by the method outlined by Ferrari and Bailey (1929) and Ferrari (1933). Twenty-gram samples of wheat meal were extracted with 100 cc. of naphtha-alcohol solvent and the transmittances of the extracts determined in a 10 cm. cell at 4358 Ängstrom, using a Bausch & Lomb spectrophotometer.

Experimental

Influence of meal granulation on the carotenoid pigments extracted. It is a well-known fact that different wheats ground on the same mill do not necessarily result in flour or meal possessing the same granulation. Coleman and Christie (1926) and Whiteside (1931) indicate the difficulties arising from this cause in preparing the sample for the colour test, and point out the importance of grinding the meal very fine. Several preliminary experiments were conducted, therefore, to study the relation of fineness of meal to carotenoid pigment extraction. Three varieties of wheat, varying widely in the amount of carotenoid pigments and in meal granulation, were ground coarse, medium, and very fine on the Labconco mill. The carotenoid pigments were then extracted from each according to the procedure already described. The results are shown in Table I.

It will be noted from the above table that the finer the granulation of the meal, the greater is the extraction of the pigments. In the soft

TABLE I

The Effect of Fineness of Whole Wheat Meal on the Extraction of Carotenoid Pigments

Variety	Condition of meal	Granulation number[1]	Carotene p.p.m.	Class
Purdue No. 1	Coarse	73	2.03	Soft
	Medium	95	2.10	"
	Very fine	128	2.24	"
Purkof	Coarse	63	2.27	Semi-hard
	Medium	66	2.37	" "
	Very fine	90	2.69	" "
Michikof	Coarse	56	2.63	Hard
	Medium	66	2.99	"
	Very fine	87	3.17	"

[1] The procedure developed by Cutler and Brinson (1935) was used to determine the granulation number.

variety (Purdue No. 1), however, the difference in the amount of carotene between the coarse and fine meal is relatively less than for the semi-hard or hard varieties. It will also be observed that even though the varieties were ground on the same mill their granulation numbers were high in the soft wheat and low in the hard wheat. This is in keeping with the findings of Cutler and Brinson (1935). In this study, except for a few semi-hard varieties, most of the 101 wheats were soft and when ground very fine gave a high granulation number.

Carotenoid pigment in wheat varieties. In order to breed wheats capable of producing flour of the desired colour, the plant breeder must first know the carotenoid content of his parental stocks. At present very little information of this nature is available. Indeed, the authors were unable to discover any such data concerning the standard varieties of soft wheat. Colour determinations were made, therefore, on some 29 winter wheat varieties, most of which would be classed as soft; a few were semi-hard. The varieites studied were those which are included in the Cooperative Uniform Winterhardiness Tests and were chosen largely because they were grown under uniform soil conditions in quadruplicate series. The varieties are arranged in the order of decreasing carotenoid content, and the data shown in Table II.

The carotenoid content, expressed as carotene parts per million, among these varieties, varied from 1.70 to 3.80 in 1933 and 1.80 to 3.80 in 1934. It is of interest to note that the five varieties on the upper end of the array, which contain the greatest amount of carotenoid pig-

TABLE II

CAROTENOID PIGMENTATION OF 29 SOFT AND SEMI-HARD WHEAT VARIETIES WHEN GROWN AT LAFAYETTE, INDIANA, DURING 1932–33 AND 1933–34

Variety	Carotene p.p.m.[1] 1933	1934	Variety	Carotene p.p.m.[1] 1933	1934
Progeny No. 2 (Ill.)	3.80	3.80	Mediterranean Sel.	2.14	2.29
Minturki	3.69	3.31	Nabob	2.17	2.25
Minhardi	3.54	3.38	Purdue No. 1	2.22	2.12
Kharkof	2.68	2.68	Currell	2.03	2.14
Purkof	2.63	2.70	Rudy	1.98	2.09
Fulhio	2.70	2.63	Fulcaster	1.95	2.12
Wisconsin Ped. No. 2	—	2.63	Forward	2.00	2.03
Honor	2.50	2.52	Leaps	2.00	—
Purplestraw	2.37	2.65	Red Rock	1.75	2.22
American Banner	2.47	2.37	Bald Rock	1.87	2.09
Gladden	2.14	2.52	Nittany	1.73	2.05
Kawvale	2.25	2.37	Michigan Amber	1.78	1.95
Trumbull	2.50	2.09	Junior No. 6	1.70	1.92
Valprize	2.11	2.39	Harvest Queen	1.75	1.80
Poole	2.12	2.37			

[1] Parts per million.

ments, are rather closely related to the hard wheats. Inter-annual correlation coefficients calculated on the colour data obtained from these varieties for two years showed ($r = +0.960 \pm .015$); when the data for the first three varieties were omitted because of their wide deviation ($r = +0.871 \pm .050$). Coefficients of this magnitude not only emphasize consistent behavior from season to season but also that meal colour as expressed in carotenoid pigments is an inherited varietal characteristic and is therefore amenable to the wheat breeder's art.

Carotenoid pigments in hybrid strains. Seventy-two strains derived from Purkof × Michigan Amber and Purkof × Fulhio crosses and carefully selected for specific soft wheat characteristics, were grown under comparable environmental conditions during 1932–33 and 1933–34 and studied for their quantitative carotenoid pigments. These data have been arranged in a correlation table, Table III.

TABLE III

CORRELATION TABLE SHOWING THE ASSOCIATION BETWEEN THE CAROTENOID CONTENT OF 72 HYBRID STRAINS WHEN GROWN DURING 1932–33 AND 1933–34

Carotene p.p.m. 1933–34 (y)	Carotene p.p.m. 1932–33 (x)											
	1.6	1.8	2.0	2.2	2.4	2.6	2.8	3.0	3.2	3.4	3.6	f
1.8	1		1									2
2.0		1	1									2
2.2			7	5								12
2.4			3	4	3	3						13
2.6				1	4	4	2	1				12
2.8							3	5	1	1		10
3.0						1		2	2	1		6
3.2								2	2		1	5
3.4								2	4	3		9
3.6											1	1
												72

$r = 0.904 \pm .020$

The data show a wide range of variation in carotenoid pigmentation among the 72 selected strains in each of the seasons 1932–33 and 1933–34. An inter-annual correlation coefficient was calculated for the purpose of learning the extent to which there was a tendency for hybrid selections of diverse nature to react the same way in different seasons. The correlation coefficient ($r = +0.904 \pm .020$) is of high

magnitude and indicates quite definitely that carotenoid pigmentation in wheat is an inherited varietal characteristic.

Carotenoid pigmentation has a genetic basis. Further evidence of the probable genetic basis of this peculiar characteristic is seen by an examination of the colour data derived from a few selected families chosen from the same crosses as referred to above. The results are shown in Table IV.

TABLE IV

HYBRID FAMILIES OF A PURKOF X MICHIGAN AMBER AND FULHIO CROSS SHOWING GENETIC DIFFERENCES AS TO CAROTENOID PIGMENTS. HARVEST CROPS OF 1933 AND 1934

Family	Carotene p.p.m.[1] 1933	1934	Family	Carotene p.p.m.[1] 1933	1934
153-5	1.66	1.85	16-5	2.19	2.12
			16-10	2.60	2.60
76-3	2.03	2.14	16-2	2.76	2.76
76-4	2.12	2.19	16-1	2.91	2.86
76-5	2.05	2.10	16-3	2.89	3.12
76-6	2.03	2.14			
63-5	2.03	2.27	13-1	3.12	3.18
63-2	2.34	2.50	13-9	3.09	3.33
63-35	2.63	2.55	13-5	3.51	3.34

[1] Parts per million.

It will be observed in Table IV that family 153–5 is very low in carotenoid pigment content, whereas the three progenies of family 13 are high. Furthermore, family 76 appears to be homozygous for this character since all four progenies have about the same amount of carotene for both years. On the other hand, families 63, 16, and 13 appear to be heterozygous since their progenies segregate consistently for varying amounts of pigments. This provides further evidence that carotenoid pigmentation in wheat is an heritable characteristic. In the light of the foregoing evidence, it seems quite clear that the breeder can breed new varieties of wheat of the desired carotenoid content.

Summary

The amount of carotenoid pigments was studied on finely ground whole wheat meal of 29 wheat varieties and 72 hybrid strains grown under uniform environmental conditions at Lafayette, Indiana, during 1932–33 and 1933–34, following the procedure outlined by Ferrari and Bailey, and Ferrari.

Granulation or fineness of the wheat meal had an influence on the

extraction of the pigments. The finer the wheat meal, the greater was the extraction of the carotenoid pigments.

Significant, positive, inter-annual, correlation coefficients for carotenoid pigments were obtained between the results of different seasons when wheats of diverse nature were grown under similar environmental conditions, indicating that carotenoid pigmentation is an inherited varietal characteristic. Hybrid families which appear to be homozygous and others heterozygous for this character are presented.

The range of the wheats studied in carotenoid pigmentation, expressed as carotene in parts per million, varied from 1.66 to 3.80 for 1932–33 to 1.80 to 3.80 for 1933–34.

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BOOK REVIEWS

Structure and Composition of Foods. Vol. II. Vegetables, Legumes, Fruits. By Andrew L. Winton and Kate Barber Winton. Published by John Wiley and Sons, Inc., New York, New York. 1935. Price $15.00.

This is a companion volume to one previously reviewed, and is truly a remarkable work. It is almost impossible to believe that so much information can be condensed into 900 pages. This could only be possible by the terse and concise presentation of essential facts. No elaborate use of words is found just in order to fill pages. The work is replete with drawings and illustrations, all the work of the authors. The illustrations generally include a drawing of the subject as found in commerce and microscopic cuts of the cross-sections of the flowers and fruit and, where essential, of the leafy portions of the plant.

Presentation of the material follows strictly a well defined plan. First is given the common and botanical names. The common names are also given in French, Spanish, Italian, and German when possible. Then follows a discussion of the use of the subject where found native, and where produced commercially. Then an excellent presentation of macroscopic features and microscopic structure with special mention of the microscopic features of the pericarp, spermoderm, perisperm, endosperm and embryo.

Next follows a discussion of the chemical composition not only of the fleshy parts of the subject, but also of the seeds and particularly where the seeds are commercially or medicinally important. Many tables of chemical composition are given which generally include water, protein, fat, N-f extract, fiber, ash and special constituents of the subject. Special attention is paid to the aromatic and flavoring ingredients of the subject as also of any toxic or medicinal properties.

Enzymes and enzymic properties are fully discussed where important as is also, the mineral constituents and many analyses of the ash of the subject are given. The final discussion deals with the minor mineral constituents. Where important, the proteins, acids, amino acids, alkaloids, glycerides, nitrogenous bases, phosphorous-organic compounds, fatty oils and sugars of the subject or its seeds are fully discussed. Of interest are the discussions and data of changes in composition during growth, ripening and storage and the chemical composition of the various parts of the subject, its fruit and seeds.

The authors do not confine themselves to the vegetables, legumes and fruits as produced commercially, but include all known to be used as food by man. Careful attention is taken to give full credit for all analyses, data and information obtained from other authors or sources.

Taken as a whole, it is a valuable and outstanding work and essential in the library of the food chemists and students and all technicians in the food industry

S. J. LAWELLIN

The Biochemistry of the Lipids. By Henry B. Bull. Published by Burgess Publishing Company, Minneapolis, Minnesota. 1935. 127 pages. Price $3.25.

Three years ago the Division of Agricultural Biochemistry of the University of Minnesota modified its general program of instruction. The survey course in biochemistry that had been given for nearly a score of years was discontinued (except for summer session classes) and in its place there was offered a series of five distinct units or courses in (1) Colloids, (2) Proteins, (3) Carbohydrates, (4) Enzymes, and (5) Lipids and Fats, respectively. The last named unit was placed in charge of Dr. Bull, who compiled a new manual for use in that connection. His notes have now been converted into a mimeographed book for the convenience of all concerned.

The book includes a discussion of the constitution, biochemical and biophysical properties of the fatty acids, soaps, sterols, fats and oils, phospholipids, glycolipids, and carbohydrate esters of the fatty acids in plant and animal tissues. The material used has been collected from a wide range of sources and has evidently been carefully selected from the standpoint of pertinence and dependability. The reviewer does not know of any text in the English language that covers so many interesting and significant fundamental facts in such conveniently compact space. It is not in any sense a handbook of the chemical and physical constants of the natural fats but instead it represents a logical attempt to discuss these properties in terms of chemical constitution and biological significance. Thus it becomes a useful approach to an understanding of the lipids, and an excellent supplement to the larger manuals which describe the individual fats.

C. H. BAILEY

Recherches sur l'Insaponifiable de l'Huile de Froment. By A. Dangoumau, Director du Laboratoire municipal de Chimie physiologique de la Ville de Bordeaux. With a preface by De M. L'Abbé Dubaquié, Directeur de la Station agronomique de Bordeaux. Published by the Librairie Delmas, Bordeaux, 1935. 148 pp., paper cover.

In the introduction the author discusses the nutritive value of wheat proteins, the minerals of wheat with emphasis on calcium and phosphorus, and the vitamins. The next section is devoted to the composition of wheat and its various milled fractions. Unsaponifiable matter is treated at length under the headings "composition of unsaponifiable matter," and "carotene and vitamin A in wheat flour." The vitamin A activity of wheat oil is illustrated by many photographs and X-ray pictures of the rat. The author expresses the opinion that the growth obtained was due not only to carotene, but to vitamin A per se.

Under the title "The Action of Irradiated Flour on Experimental Rickets" a general review of the literature is given followed by experiments with irradiated flour. Ultra violet irradiation for as little as five minutes with a "Gallois" burner appears to impart antirachitic properties to a commercial flour that replaces the flour in a vitamin D deficient diet. The study is thorough, embracing biological assays, X-ray pictures of the bones, histological studies, chemical analyses of the bones, and photomicrographs. The author expresses the opinion that it is not necessary or desirable for irradiated foods to have vitamin potency comparable to medicinal products, but believes it is advantageous to augment in this way ordinary foods deficient in vitamin D.

Then follows a section devoted to a study of the sterols of wheat, introduced by a review of the literature. The author identifies the sterols as ergosterol, and two combined sterols, namely, sitosteryl palmitate and phytosteroline—a sitosterol glucoside. The latter finding confirms the work of Nakamura and Schiba.

In conclusion a study of the crystalline form of sitosterol, sitosteryl palmitate and palmitic acid is undertaken, illustrated by many excellent photomicrographs. The author's treatment of this section is very interesting and in some respects unique.

CHARLES G. FERRARI
A. B. CROZE

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